REPORT DOCUMENTATION PAGE

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. **PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

1. REPORT DATE (DD-MM-YYYY) 16-07-2003	2. REPORT TYPE Technical Paper	3. DATES COVERED (From - To)

4. TITLE AND SUBTITLE

Preparation and Characterization of the Argentates: $[AG\{P(CF_3)_2\}_2]^-$, $[Ag\{\mu\text{-}P(CF_3)_2M(CO)_5\}_2]^-$ (M = Cr, W) and $[Ag\{(\mu\text{-}P(C_6F_5)_2)W(CO)_5\}_2]^-$: X-ray Crystal Structure of $[K(18\text{-crown-6}][Ag\{(\mu\text{-}\{(CF_3)_2)Cr(CO)_5\}_2]$

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

Berthold Hoge, Christoph Thösen, Tobias Herrmann (all Univ zu Köln); Ashwani Vij (AFRL/PRSP)

5d. PROJECT NUMBER
DARP

5e. TASK NUMBER
A205

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/PRSP
10 East Saturn Blvd.
Edwards AFB CA 93525-7680

8. PERFORMING ORGANIZATION REPORT NUMBER

AFRL-PR-ED-TP-2003-192

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/PRS
5 Pollux Drive
Edwards AFB CA 93524-7048

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S NUMBER(S)
AFRL-PR-ED-TP-2003-192

12. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

For presentation in the Journal of Inorganic Chemistry.

14. ABSTRACT

20030812 220

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:

a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Leilani Richardson
Unclassified	Unclassified	Unclassified	A	53	19b. TELEPHONE NUMBER (include area code) (661) 275-5015

Synopsis

Preparation and Characterization of the Argentates:
$[Ag\{P(CF_3)_2\}_2]^-$, $[Ag\{(\mu\text{-}P(CF_3)_2)M(CO)_5\}_2]^-$ (M = Cr, W)
and $[Ag\{(\mu\text{-}P(C_6F_5)_2)W(CO)_5\}_2]^-$: X-ray Crystal Structure
of $[K(18\text{-crown-}6)][Ag\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2]$

Berthold Hoge,[a*] Christoph Thösen,[a] Tobias Herrmann,[a] and
Ashwani Vij[b]

[a] *Institut für Anorganische Chemie, Universität zu Köln, D-50939
Köln, Germany and Air Force Research Laboratory
(AFRL/PRS), Space and Missile Propulsion Division,
Edwards Air Force Base, California 93524.*
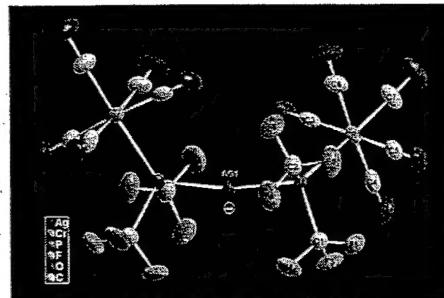
The novel bis{bis(trifluoromethyl)phosphanido}argentate,
$[Ag\{P(CF_3)_2\}_2]^-$, is obtained *via* the reaction of $HP(CF_3)_2$
with $[Ag(CN)_2]^-$ and isolated as its [K(18-crown-6)] salt.
NMR spectroscopic evidence for the $[Ag\{P(CF_3)P(CF_3)_2\}_2]^-$
ion is also obtained in this study. Treatment of the
phosphane complexes $[M(CO)_5PH(CF_3)_2]$ (M = Cr, W) with
$[K(18\text{-crown-}6)][Ag(CN)_2]$ yields trinuclear argentates,
$[Ag\{(\mu\text{-}P(CF_3)_2)M(CO)_5\}_2]^-$ (M = Cr or W). In [K(18-
crown-6)][Ag\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2]$, the C_2 symmetric
anion, $[Ag\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2]^-$, shows a nearly linear
arrangement of the P-Ag-P unit. This is the first example of
a structurally characterized perfluorinated phosphanido-
metal complex.

Preparation and Characterization of the Argentates: $[Ag\{P(CF_3)_2\}_2]^-$, $[Ag\{(\mu$-$P(CF_3)_2)M(CO)_5\}_2]^-$ (M = Cr, W) and $[Ag\{(\mu$-$P(C_6F_5)_2)W(CO)_5\}_2]^-$: X-ray Crystal Structure of $[K(18$-crown-$6)][Ag\{(\mu$-$P(CF_3)_2)Cr(CO)_5\}_2]$

Author for correspondence:

Dr. B. Hoge

Institut für Anorganische Chemie

Universität zu Köln

Greinstr. 6

D-50939 Köln, Germany

FAX: 049-221-470-5196

E-Mail: b.hoge@uni-koeln.de

Preparation and Characterization of the Argentates: $[Ag\{P(CF_3)_2\}_2]^-$, $[Ag\{(\mu$-$P(CF_3)_2)M(CO)_5\}_2]^-$ (M = Cr, W) and $[Ag\{(\mu$-$P(C_6F_5)_2)W(CO)_5\}_2]^-$: X-ray Crystal Structure of $[K(18$-crown-$6)][Ag\{(\mu$-$P(CF_3)_2)Cr(CO)_5\}_2]$

Berthold Hoge,[a*] Christoph Thösen,[a] Tobias Herrmann,[a] and Ashwani Vij[b]

[a] *Institut für Anorganische Chemie, Universität zu Köln, D-50939 Köln, Germany*

[b] *Air Force Research Laboratory (AFRL/PRS), Space and Missile Propulsion Division, Edwards Air Force Base, California 93524. email ashwani.vij@edwards.af.mil*

Abstract

The novel bis{bis(trifluoromethyl)phosphanido}argentate, $[Ag\{P(CF_3)_2\}_2]^-$, is obtained via the reaction of $HP(CF_3)_2$ with $[Ag(CN)_2]^-$ and isolated as its [K(18-crown-6)] salt. If the cyclic phosphane $(PCF_3)_4$ is reacted with a slight excess of $[K(18$-crown-$6)][Ag\{P(CF_3)_2\}_2]$ a selective insertion of one PCF_3 unit into each silver phosphorus bond is observed, giving NMR spectroscopic evidence for the $[Ag\{P(CF_3)P(CF_3)_2\}_2]^-$ ion.

On treatment of the phosphane complexes $[M(CO)_5PH(CF_3)_2]$ (M = Cr, W) with $[K(18$-crown-$6)][Ag(CN)_2]$ the formation of the comparable trinuclear argentates, $[Ag\{(\mu$-$P(CF_3)_2)M(CO)_5\}_2]^-$ is achieved. The chromium compound $[K(18$-crown-$6)][Ag\{(\mu$-$P(CF_3)_2)Cr(CO)_5\}_2]$ crystallizes in a non-centrosymmetric space group $Fdd2$ (no. 43), $a = 2970.2(6)$ pm, $b = 1584.5(3)$ pm, $c = 1787.0(4)$, $V = 8.410(3)$ nm^3, $Z = 8$. The C_2 symmetric anion, $[Ag\{(\mu$-$P(CF_3)_2)Cr(CO)_5\}_2]^-$, shows a nearly linear arrangement of the P-Ag-P unit and represents the first example of a structurally characterized perfluorinated phosphanido-metal complex. Although, the bis(pentafluorophenyl)phosphanido compound $[Ag\{P(C_6F_5)_2\}_2]^-$ could not be obtained so far, the synthesis of its trinuclear counterpart, $[K(18$-crown-$6)][Ag\{(\mu$-$P(C_6F_5)_2)W(CO)_5\}_2]$ was successful.

Introduction

In our field of research, nucleophilic bis(perfluoroorganyl)phosphanide sources are essential for the synthesis of chiral, bidentate strong π-acidic bis(perfluoroorganyl)phosphane ligands. The bis(trifluoromethyl)phosphanide ion exhibits negative hyperconjugation which results in a C-F activation and therefore, favors a decomposition of this compound in solution.

To obtain selectively nucleophilic $P(CF_3)_2$ group transfer reagents, it is essential to reduce the negative hyperconjugation by addition of a weak Lewis acid. Terminal bis(perfluoroorganyl)phosphanido transition metal complexes are potential nucleophilic $P(CF_3)_2$ group transfer reagents.[1, 2, 3]

To the best of our knowledge, no binary bis(phosphanido)argentate of the general formula $[Ag(PR_2)_2]^-$ (R = noncyclic univalent group) is known so far. Only the trinuclear counterparts $[Ag\{(\mu\text{-}PR_2)M(CO)_5\}_2]^-$ (R = Ph, C_6H_{11} and M = Cr, Mo, W) have been characterized by ^{31}P NMR spectroscopy and some of them by elemental analysis.[4] A novel gold complex, $[Ag\{(\mu\text{-}PPh_2)Au(C_6F_5)_5\}_2]^-$ has also been structurally characterized (Blanco, M. C.; Fernandez, E. J.; Jones, P. G.; Laguna, A.; Lopez-de-Luzuriaga, J. M.; Olmos, M. E. *Angew. Chem. Intl. Ed. Engl.*, **1998**, *37*, 3042.)

Experimental Section

Materials and Apparatus. Chemicals were obtained from commercial sources and used without further purification. Literature methods were used for the synthesis of $HP(C_6F_5)_2$, $[W(CO)_5PH(C_6F_5)_2]$, $[W(CO)_5PH(CF_3)_2]^5$ and $[Cr(CO)_5PH(CF_3)_2]$.[6] Solvents were purified by standard methods.[7] Standard high-vacuum techniques were employed throughout all preparative procedures; nonvolatile compounds were handled in a dry N_2 atmosphere by using Schlenk techniques.

Infrared spectra were recorded on a Nicolet-5PC-FT-IR spectrometer as KBr pellets. Raman spectra were measured on a Bruker FRA-106/s spectrometer with a Nd:YAG laser operating at $\lambda = 1064$ nm.

The NMR spectra were recorded on Bruker Model AMX 300 (^{31}P, 121.50 MHz; ^{19}F, 282.35 MHz; ^{13}C 75:47 MHz) and Bruker AC200 spectrometers (^{31}P, 81.01 MHz; ^{19}F 188.31 MHz; ^{1}H, 200.13 MHz). Fluorine decoupled phosphorus spectra were measured on a Bruker DRX 500 spectrometer (^{31}P, 202.40 MHz) with positive shifts being downfield from the external standards (85% orthophosphoric acid (^{31}P), CCl$_3$F (^{19}F) and TMS (^{1}H)). Higher order NMR spectra were calculated with the program gNMR.[8]

Single crystal x-ray diffraction studies were performed at 170(2) K using a STOE IPDS I diffractometer using graphite monochromated MoKα ($\lambda = 71.073$ pm). Details of the structural parameters are listed in Table 1 and selective bond lengths, bond angles and torsion angles are listed in Table 2. A complete summary of structure solution and refinement is described in the *cif* file deposited with this journal. The structure was solved in space group *Fdd2* using Patterson Method and refined with SHELXTL V6.10 (SHELXTL 6.12 for Windows, Bruker AXS Inc. Madison, Wi. USA). The positions of silver and potassium atoms were located on special position (2 fold axis) and the chromium atom was found to occupy a general position. Further structure expansion revealed the remaining atoms of the [Ag{(P(CF$_3$)$_2$)Cr(CO)$_5$}]$^-$ anion. Since the silver atom sits on a special position, the complete anion could be generated using the symmetry operation (3/2 – x, ½ - y, z). From a disordered array of electron density around the potassium atom in the shape of "half-fan", six oxygen positions were identified, which constituted the two-component half-crown ether structure. Two terminal carbon atoms of this half-crown configuration, later named C12 and C18, were subsequently located and were found to occupy special positions. The two-component disordered 18-crown-6 was then generated using the symmetry operation (3/2 - x, 3/2 - y, +z).

One of these components were retained and used for further refinement after moving C12 and C18 off the special position and refining the symmetry generated equivalents with an occupancy factor of 0.5 using the PART -1 command in SHELXL . During this refinement, the C-C and C-O distances of the 18-crown-6 moiety were restrained to be equal with maximum allowable deviation of 1 pm. Similarly, thermal parameters of the disordered atoms were also restrained using SIMU and DELU commands. Finally, all non-hydrogen atoms were refined anisotopically and hydrogen atoms were added to the carbon atoms at calculated positions. Since the structure belongs to a non-centrosymmetric space group *Fdd2*, the absolute configuration was determined by refining the Flack's parameter to 0.04(2), which confirms the corrected "handedness" of the molecular structure.

Preparation of [K(18-crown-6)][Ag{P(CF$_3$)$_2$}$_2$]. A 1.75 (10.28 mmol) amount of HP(CF$_3$)$_2$ was condensed on a solution of 1.89 g (4.08 mmol) of [K(18-crown-6)][Ag(CN)$_2$] in 10 mL of acetone at -196 °C. After stirring the mixture at -78 °C for 1 hour, the temperature was allowed to rise to room temperature and the solution was evaporated to dryness. The residue was extracted several times with hexane, yielding 2.45 g (3.27 mmol, 80%) of [K(18-crown-6)][Ag{P(CF$_3$)$_2$}$_2$] as a colorless solid. Elemental anal. (calc. for C$_{16}$H$_{24}$AgF$_{12}$KO$_6$P$_2$): C 25.50 (25.65); H 3.42 (3.23). Infrared spectrum (cm^{-1}, KBr pellet): 3854 vw, 3445 w, 2891 s, 2829 m, 2748 vw, 1720 w, 1475 m, 1456 m, 1435 w, 1354 s, 1286 m, 1251 s, 1163 vs, 1109 vs, 962 s, 839 m, 734 vw, 559 w, 530 w, 459 m. Raman (cm^{-1}): 2956 (2), 2917 (5), 2889 (100), 2848 (27), 2811 (13), 2150 (20), 2136 (6), 1476 (44), 1274 (37), 1246 (11), 1183 (7), 1146 (17), 1068 (10), 873 (30), 832 (18), 736 (64), 549 (15), 534 (2), 465 (22), 294 (13), 275 (36), 240 (5), 140 (4), 105 (29). Negative ESI mass spectrum (acetone; 10^{-3} M) {m/z (%) [assignment]}: 999 (38) [Ag$_3$\{P(CF$_3$)$_2$\}$_4$]$^-$, 723 (100) [Ag$_2$\{P(CF$_3$)$_2$\}$_2$]$^-$, 573 (18) [Ag$_2$\{P(CF$_3$)$_2$\}$_2$F]$^-$, 445 (35) [Ag\{P(CF$_3$)$_2$\}$_2$]$^-$. NMR data (THF; 298 K): δ(^{31}P) 2.8 ppm; δ(^{19}F) -39.4 ppm; δ(^{13}C) 138.0 ppm; ^1J(CF) 317 Hz; ^2J(PF) 57.9 Hz;

^1J(PC) 49 Hz; ^3J(CF) 9 Hz; ^4J(FF) 8 Hz. The NMR data of [K(18-crown-6)]$^+$ are unaffected by the counterion ($\delta(^{13}C)$ 71 ppm, $\delta(^1H)$ 3 ppm).

Preparation of [K(18-crown-6)][Ag{(μ-P(CF$_3$)$_2$)W(CO)$_5$}$_2$]. 1.06 g (2.14 mmol) of [W(CO)$_5$PH(CF$_3$)$_2$] and 0.48 g (1.02 mmol) of [K(18-crown-6)][Ag(CN)$_2$] were dissolved in 5 mL of DMF and stirred for two hours at room temperature. After evaporation of the green solution, the residue was extracted with diethyl ether giving 0.99 g (0.71 mmol, 70%) of [K(18-crown-6)][Ag{(μ-P(CF$_3$)$_2$)W(CO)$_5$}$_2$] as a green powder. TG analysis: decomposition at 250 °C. Elemental analysis (calc. for C$_{26}$H$_{24}$AgF$_{12}$KO$_{16}$P$_2$W$_2$): C 22.63 (22.35); H 1.78 (1.73); N 0.15 (0.00). Infrared spectrum (cm^{-1}, KBr pellet): 3435 m, 2914 w, 2072 m, 2017 w, 1981 m, 1946 vs, 1931 vs, 1903 vs, 1903 vs, 1630 w, 1475 vw, 1456 vw, 1353 w, 1286 vw, ·1251 w, 1168 s, 1110 s, 1086 m, 963 w, 841 vw, 598 m, 578 m, 469 w, 459 w, 428 w. Raman (cm^{-1}, as a result of fluorescence, some of the intensities are uncertain): 2951 (8), 2918 (12), 2893 (10), 2879 (10), 2847 (8), 2071 (60), 1979 (100), 1917 (50), 1475 (5), 974 (20) 958·(20), 870, 798,·737, 449, 428 (40), 405, 349, 331, 222, 94 (60). Negative ESI mass spectrum (acetone; 10^{-3} M) {m/z (%) [assignment]}: 1094 (100) [Ag{P(CF$_3$)$_2$W(CO)$_5$}$_2$]$^-$. NMR data (CDCl$_3$/DMF; 298 K): $\delta(^{31}P)$ 13.8 ppm; $\delta(^{19}F)$ -48.5 ppm; $^1J(^{109/107}AgP)$ 424.6/369.4 Hz; $^1J(^{183}WP)$ 155.6 Hz; ^2J(PP) 100.7 Hz; ^2J(PF) 63.3 Hz; ^4J(PF) 2.5 Hz.

Preparation of [K(18-crown-6)][Ag{(μ-P(C$_6$F$_5$)$_2$)W(CO)$_5$}$_2$]. A solution of 2.00 g (2.90 mmol) of [W(CO)$_5$PH(C$_6$F$_5$)$_2$] in 5 mL of acetone was added dropwise to a solution of 0.55 g (1.18 mmol) [K(18-crown-6)][Ag(CN)$_2$] in 10 mL of acetone at -50 °C. After allowing the temperature to rise to room temperature, the reaction mixture was evaporated to dryness. The product [K(18-crown-6)][Ag{(μ-P(C$_6$F$_5$)$_2$)W(CO)$_5$}$_2$] (1.09 g, 0.61 mmol, 50%) was obtained as a yellow powder after recrystallization from diethyl ether/hexane. TG analysis: decomposition at 180 °C. Elemental analysis (calc. for C$_{46}$H$_{24}$AgF$_{20}$KO$_{16}$P$_2$W$_2$): C 31.71 (30.88); H 1.84 (1.35). Infrared spectrum (cm^{-1}, KBr pellet): 2918 m, 2880 w, 2930 vw, 2066

s, 1977 s, 1937 vs, 1921 vs, 1887 s, 1638 w, 1624 vw, 1514 s, 1468 s, 1379 vw, 1352 w, 1284 w, 1250 w, 1111 s, 1080 s, 972 s, 837 w, 826 w, 760 vw, 750 vw, 725 vw, 634 w, 598 m, 577 m, 532 vw, 503 vw, 426 vw, 413 vw. Raman (cm^{-1}): 2954 (8), 2920 (12), 2895 (11), 2848 (10) 2810 (7), 2067 (33), 1971 (100), 1944 (23), 1930 (25), 1903 (50), 1639 (22), 1514 (5), 1475 (9), 1381 (12), 1275 (8), 1246 (4), 1141 (5), 873 (4), 827 (15), 584 (19), 503 (10), 457 (30), 432 (28), 387 (10), 345 (8), 332 (6), 281 (4), 102 (60). Positive ESI mass spectrum (acetone/THF; 10^{-3} M) {m/z (%) [assignment]}: 303 (100) [K(18-crown-6)]$^+$. Negative ESI mass spectrum (acetone/THF; 10^{-3} M) {m/z (%) [assignment]}: 1486 (1) $[AgP_2(C_6F_5)_4W_2(CO)_{10}]^+$, 1403 (18) $[AgP_2(C_6F_5)_4W_2(CO)_7]^+$, 1374 (6) $[AgP_2(C_6F_5)_4W_2(CO)_6]^+$, 1346 (26) $[AgP_2(C_6F_5)_4W_2(CO)_5]^+$, 1318 (8) $[AgP_2(C_6F_5)_4W_2(CO)_4]^+$, 1290 (100) $[AgP_2(C_6F_5)_4W_2(CO)_3]^+$, 1262 (7) $[AgP_2(C_6F_5)_4W_2(CO)_2]^+$, 1234 (36) $[AgP_2(C_6F_5)_4W_2(CO)]^+$, 1206 (95) $[AgP_2(C_6F_5)_4W_2]^+$, 1088 (10) $[AgP_2(C_6F_5)_4W(CO)_2]^+$, 1022 (9) $[AgP_2(C_6F_5)_4W]^+$. NMR data (CDCl$_3$; 298 K): $\delta(^{31}P)$ -126.5 ppm; $\delta(^{19}F_o)$ -128.0 ppm (m, 2F); $\delta(^{19}F_m)$ -162.8 ppm (m, 2F); $\delta(^{19}F_p)$ -156.3 ppm (m, 1F); $^1J(^{109/107}AgP)$ 437.4/380.4 Hz; $^1J(^{183}WP)$ 167.7 Hz; $^2J(PP)$ 142.4 Hz; $^3J(PF)$ 9.0 Hz; $^4J(PF)$ 8.4 Hz.

Preparation of [K(18-crown-6)][Ag{(μ-P(CF$_3$)$_2$)Cr(CO)$_5$}$_2$]. A solution of [K(18-crown-6)][Ag(CN)$_2$] in acetone was treated with a slight excess of [Cr(CO)$_5$PH(CF$_3$)$_2$] at -30 °C. After allowing the temperature to rise to room temperature, the reaction mixture was evaporated to dryness. The red oily product, [K(18-crown-6)][Ag{(μ-P(CF$_3$)$_2$)Cr(CO)$_5$}$_2$], was extracted several times with hexane. Colorless single crystals were obtained after slow condensation of hexane onto a diethyl ether solution at -45 °C. Infrared spectrum (cm^{-1}, THF solution, 1700-2400 cm^{-1}): 2064 m, 1946 vs, 1926 s. NMR data (CDCl$_3$; 298 K): $\delta(^{31}P)$ 64.8 ppm; $\delta(^{19}F)$ -45.5 ppm; $^1J(^{109/107}AgP)$ 412/359 Hz; $^2J(PP)$ 94 Hz; $^2J(PF)$ 55 Hz; $^4J(PF)$ 3 Hz.

Results and Discussion

Based on the successful synthesis of bis(perfluoroorganyl)phosphanide salts[9] and bis(perfluoroorganyl)phosphanido complexes,[2, 10] by reacting bis(perfluoroorganyl)phosphanes, $HP(R_F)_2$ ($R_F = CF_3$, C_6F_5) with cyanide compounds, we further investigated the reactions of $HP(CF_3)_2$, $HP(C_6F_5)_2$ and their comparable $M(CO)_5$ ($M = Cr$, W) complexes with dicyanoargentates.

To obtain dicyanoargentate salts that are soluble in most organic solvents, we first reacted $K[Ag(CN)_2]$ with 18-crown-6 in methanol. The product $[K(18\text{-crown-6})][Ag(CN)_2]$ was isolated as a colorless solid in a quantitative yield after evaporating the solvent.

$$K[Ag(CN)_2] + 18\text{-crown-6} \longrightarrow [K(18\text{-crown-6})][Ag(CN)_2] \qquad (1)$$
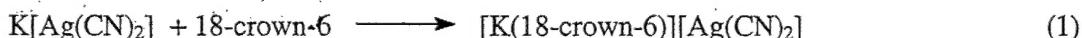
On treatment of a $[K(18\text{-crown-6})][Ag(CN)_2]$ acetone solution with a slight excess of $HP(CF_3)_2$, a complete conversion to $[Ag\{P(CF_3)_2\}_2]^-$ is achieved.

$$[Ag(CN)_2]^- + 2\,HP(CF_3)_2 \longrightarrow 2\,HCN + [Ag\{P(CF_3)_2\}_2]^- \qquad (2)$$
$$cat = [K(18\text{-crown-6})]^+$$
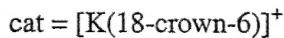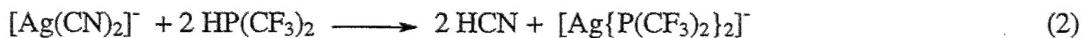
Removal of all the volatile components at room temperature, and extracting the residue several times with hexane, gives $[K(18\text{-crown-6})][Ag\{P(CF_3)_2\}_2]$ in a 80% yield. An analysis of the Raman spectrum indicates the presence of cyano compounds minor impurities. To our knowledge, the bis(bis(trifluoromethyl)phosphanido)argentate is the first isolated example of a binary di(phosphanido)argentate salt containing a perfluorinated phosphanido group.

The use of the $[Ag\{P(CF_3)_2\}_2]^-$ ion in nucleophilic displacement reactions leads to disappointing results. On treatment of $[K(18\text{-crown-6})][Ag\{P(CF_3)_2\}_2]$ solutions with ethyl iodide, the expected product $C_2H_5P(CF_3)_2$ could be obtained only in minor amounts (< 5%) even after stirring for several hours at room temperature in different solvents. Further extension of the reaction time or raising the reaction temperature above room temperature favored the formation of a number of unidentified side products.

It was found that the reaction of cyclic phosphane $(PCF_3)_4$[11] is with a slight excess of $[K(18\text{-crown-6})][Ag\{P(CF_3)_2\}_2]$ is selective and proceeds with the formation of the $[Ag\{P(CF_3)P(CF_3)_2\}_2]^-$ ion, as evidenced by NMR spectroscopic studies.

$$[Ag\{P(CF_3)_2\}_2]^- + \tfrac{1}{2}\,(PCF_3)_4 \longrightarrow \begin{bmatrix} \end{bmatrix}^- \qquad (3)$$
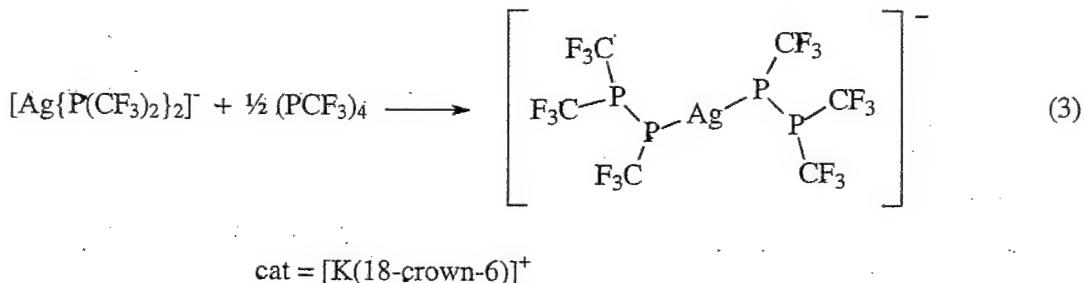
$$cat = [K(18\text{-crown-6})]^+$$

A comparable nucleophilic cleavage of the phosphorus-phosphorus bond of cyclic $(PCF_3)_4$ is observed in reactions with $[K(18\text{-crown-6})]P(CF_3)_2$, allowing the NMR spectroscopic characterization of the novel tris(trifluoromethyl)diphosphanide ion, $P(CF_3)P(CF_3)_2^-$.

$$P(CF_3)_2^- + \tfrac{1}{4}\,(PCF_3)_4 \longrightarrow \begin{bmatrix} \end{bmatrix}^- \qquad (4)$$
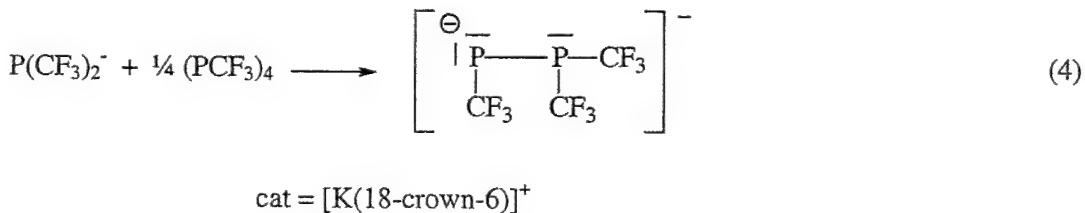
$$cat = [K(18\text{-crown-6})]^+$$

Both diphosphanide compounds (cf. eq. (3) and (4)) are generated in THF at -30 °C in selective reactions. The neat [K(18-crown-6)] salts, which are expected to be stable at room

temperature, could not be isolated so far. The NMR spectroscopic data of both anions are summarized in Table 3 and compared to the data of the starting material, the $[Ag\{P(CF_3)_2\}_2]^-$ and $P(CF_3)_2^-$ ions and the cyclic phosphane, $(PCF_3)_4$.

The ^{31}P NMR resonances of the phosphanide units of the $P(CF_3)P(CF_3)_2^-$ ion (cf. Table 3) and $[Ag\{P(CF_3)P(CF_3)_2\}_2]^-$ ions exhibit the expected shift to higher field with respect to $(PCF_3)_4$ of about 17 and 5 ppm, respectively.[12] The ^{31}P NMR resonances of the phosphane units (cf. Table 3) are shifted of about 110 and 97 ppm, respectively, to lower field in the same comparison. These characteristic shifts allow an unambiguous assignment.

The $^1J(PP)$ coupling constants of 340 and 287 Hz for the $P(CF_3)P(CF_3)_2^-$ and $[Ag\{P(CF_3)P(CF_3)_2\}_2]^-$ ions, respectively, exhibit both a strong increased magnitude with respect to the coupling of 101 Hz for $(PCF_3)_4$.[12] Both values are in the expected range and reduced by about 55 and 110 Hz, respectively, with respect to the 1,2-diethylphenyldiphosphanide ion, $P(C_2H_5)P(C_2H_5)(C_6H_5)^-$, which has been synthesized reacting $LiP(C_2H_5)(C_6H_5)$ and $(PC_2H_5)_4$.[13]

On the first view, the unexpected low nucleophilicity of the $[Ag\{P(CF_3)_2\}_2]^-$ ion in comparison to $[Hg\{P(CF_3)_2\}_2]$, which reacts smoothly to $CH_3CH_2P(CF_3)_2$ on treatment with ethyl iodide, may be surprising. As a result of the much stronger nucleophilicity of the bis(trifluoromethyl)argentate, $[Ag(CF_3)_2]^-$, with respect to $[Hg(CF_3)_2]$ we expected the comparable phosphanido argentate, $[Ag\{P(CF_3)_2\}_2]^-$, to be an even stronger nucleophilic agent.

The negative electrospray ionization (ESI) mass spectrum of a diluted acetone solution of $[K(18\text{-crown-}6)][Ag\{P(CF_3)_2\}_2]$ provides an explanation for the low nucleophilicity of the argentate ion. Besides the monometallic ion, $[Ag\{P(CF_3)_2\}_2]^-$, with a relative intensity of 35% we detected a bimetallic and a trimetallic fragment ion. The fragment ions $[Ag_2\{P(CF_3)_2\}_3]^-$ and $[Ag_3\{P(CF_3)_2\}_4]^-$ are detected with relative intensities of 100 and 38%, respectively and

exhibit the expected isotopic pattern for a bi- and trinuclear silver compound. The observation

of these higher nucleated silver fragment ions is a strong hint for an association of the

$[Ag\{P(CF_3)_2\}_2]^-$ ion in solution.

In the case of an association *via* bridging μ-phosphanido groups, the formal phosphorus

lone electron pairs are no longer available for nucleophilic displacement reactions.

The fast ligand exchange of the $[Ag\{P(CF_3)_2\}_2]^-$ ion in solution, with respect to the NMR

time scale, additionally supports an association of the argentate ion, involving bridging μ-

phosphanido groups, by the formation of oligomeric moieties:

$$
\begin{array}{c}
F_3C \quad CF_3 \\
P \\
(CF_3)_2P \!\!-\!\! Ag \quad Ag \!\!-\!\! P(CF_3)_2 \\
P \\
F_3C \quad CF_3
\end{array}
\qquad (5)
$$

E. g.
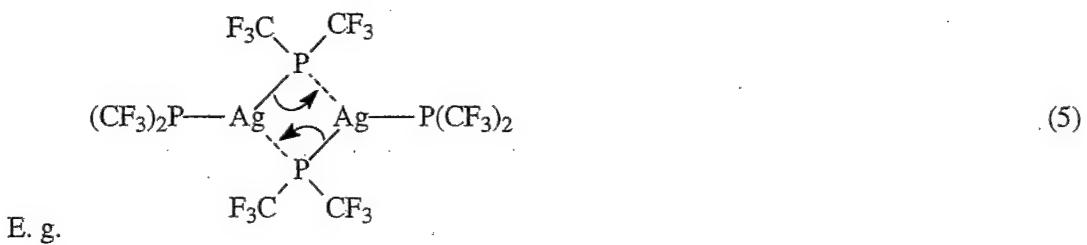
Fenske and co-workers provided structural evidence for the formation of bridging μ_2-

phosphanido groups in the polymetallic mixed phosphanido phosphane silver complexes.[14]

While the room temperature NMR spectra of the $[Ag\{P(CF_3)_2\}_2]^-$ ion exhibit sharp ^{19}F and

^{31}P NMR resonances, the low temperature NMR spectra (THF; -90 °C) exhibit very complex

spin systems, which could not be analyzed so far. The room temperature ^{19}F and ^{31}P NMR

spectra show line widths of less than 4 Hz but show no hints for fluorine or phosphorus silver

nuclear couplings. To obtain NMR spectroscopic evidence for the formation of a silver

phosphorus bond *via* the detection of the ^{31}P nuclear couplings with the two NMR active

silver isotopes, [109]Ag and [107]Ag, it was necessary to slow down the ligand exchange process.

The coordination of each phosphanido ligand of an $[Ag\{P(CF_3)_2\}_2]^-$ ion to a $M(CO)_5$

moiety will prevent the involvement of the formal phosphorus lone pairs in any kind of

- 12 -

association of the $[Ag\{P(CF_3)_2\}_2]^-$ moieties. As a consequence, this "blockade" of the phosphorus lone pairs should slow down the ligand exchange process, allowing a detection of the phosphorus silver nuclear couplings.

The reactions of $[W(CO)_5PH(CF_3)_2]^6$ or $[Cr(CO)_5PH(CF_3)_2]^6$ with [K(18-crown-6)][Ag(CN)_2] proceed under formation of the corresponding trinuclear argentates, $[Ag\{(\mu-P(CF_3)_2)M(CO)_5\}_2]^-$ (M = W and Cr) in selective reactions at -50 °C in acetone.
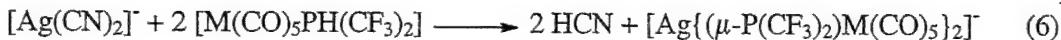
$$[Ag(CN)_2]^- + 2\ [M(CO)_5PH(CF_3)_2] \longrightarrow 2\ HCN + [Ag\{(\mu-P(CF_3)_2)M(CO)_5\}_2]^- \quad (6)$$
$$M = Cr,\ W;\ cat = [K(18\text{-crown-}6)]^+$$

After the removal of all the volatiles *in vacuo* at room temperature and extracting the residue once with diethyl ether, the product $[K(18\text{-crown-}6)][Ag\{(\mu-P(CF_3)_2)W(CO)_5\}_2]$ was obtained as a green powder. The trinuclear argentate ion, $[Ag\{(\mu-P(CF_3)_2)W(CO)_5\}_2]^-$, exhibits no evidence for an association in solution. The ESI mass spectrum reveals only one fragment at m/z = 1094 assigned to the $[Ag\{(\mu-P(CF_3)_2)W(CO)_5\}_2]^-$ ion.

Colorless crystals of the chromium compound, $[K(18\text{-crown-}6)]^+ [Ag\{(\mu-P(CF_3)_2)Cr(CO)_5\}_2]^-$ were obtained by slow condensation of hexane onto a saturated diethyl ether solution at -45 °C. This compound crystallizes in a non-centrosymmetric space group *Fdd*2. The asymmetric unit shows the silver atom on a two-fold axis with only half of the anionic structure. The complete C_2 symmetric structure of $[Ag\{(\mu-P(CF_3)_2)Cr(CO)_5\}_2]^-$ anion is generated using a symmetry operation ($a = 3/2 - x$, ½ - y, z) yielding P1^a-Ag1-P1 bond angle of 169.17(6)°, Ag-P distance of 239.1(1) pm and P'-Ag-P-Cr torsion angle of 39.02(4)° (Fig. 1). A survey of the CCSD (Conquest 1.5, Cambridge Crystal Structure Database, CCDC, Cambridge, U.K.) reveals an isolated example for an analogous Ag(I) anion, $[Ag\{(\mu-P(C_6H_5)_2)Au(C_6F_5)_3\}_2]^-$, which contains a linear P-Ag-P bond (180°) with Ag-P distance of

- 13 -

P distance of 238.6 pm. This distance is in the range of 238-241 pm found in other linear or ring P-Ag-P linked complexes. The Cr-P distance of and 238.9(1) pm is shorter than that found in $[(CO)_5CrP(CF_3)(C_2H_5OCN(CH_3)_2]$ (246.9 pm) (Grobe, J.; le Van, D.; Althoff, U.; Krebs, B.; Dartmann, M.; Gleiter, R. *Heteroatom. Chem.* **1991**, *2*, 385).

The potassium atom lies on a 2-fold axis and is encapsulated by a severely disordered 18-crown-6 ring. The disordered component of the crown ether is generated by symmetry (3/2 − x, 3/2 − y, z) (see experimental section) and the two conformations are rotated by ~22.4° with respect to each other as shown in Fig. 2. An analysis of the mean plane formed by the oxygen atoms of the crown ether show a near-planar arrangement with deviation of 19.3(5) pm with the potassium atom almost coplanar at 2.6(3) pm with this mean plane. The maximum deviations from the mean plane described are shown by O8 and O9 atoms +22.5(5) and -24.8(5) pm, respectively.

In addition to the hexacoordination from oxygen atoms of the 18-crown-6 ring, the potassium atom further coordinates to one fluorine atom (F5) of the $P(CF_3)_2$ group (K⋯F = 3.052(5) pm) and one oxygen atom (O3) of the $Cr(CO)_5$ (K⋯O = 3.287(3)pm), as well as their symmetry equivalents O3[b] and F5[b] (b = 3/2 − x, 3/2 − y, z). These distances are shorter than the sum of van der Waal radii of K and O (427 pm), and K and F (422 pm) and result in the formation of a seven-membered ring which increases the coordination number of potassium to ten. Only two K[18-crown-6] structures containing both K⋯O and K⋯F contacts could be found in CCSD. For these compounds, the K⋯O and K⋯F distances lie in the range 267.6-288.0 pm and 289.5-333.1 pm, respectively (Kawashima, T.; Iwama, N.; Tokitoh, N.; Okazaki, R. *J. Org. Chem.* **1994**, *59*, 491. Kawashima, T.; Nishiwaki, Y.; Okazaki, R. *J. Organomet. Chem.* **1995**, *499*, 143.) Further, in these oxagermetanide anion containing 18-crown-6 complexes, the potassium atom lies 15 (Kawashima, T.; Iwama, N.; Tokitoh, N.;

- 14 -

Okazaki, R. *J. Org. Chem.* **1994**, *59*, 491) and 121 (Kawashima, T., Nishiwaki, Y., Okazaki, R. *J. Organomet. Chem.* **1995**, *499*, 143.) pm above the crown ring.

The crystal packing diagram, shown in Fig. 3, shows the formation of two Ag···O5 contacts at 321.9(5) pm, which is comparable to the sum of van der Waal radii for Ag and O (324 pm). The O-Ag-O bond is almost linear at 169.4° and is twisted by 81.9° with respect to P1^a-Ag1-P1 bond. These Ag-O and K-O/F result in the formation of a zig-zag polymeric chain along the *a*- and *b*-axis, respectively (Fig. 3).

As a result of slowing down the ligand exchange process, the silver phosphorus coupling constants $^1J(^{109/107}$AgP) could be determined in the ^{31}P NMR spectrum to 425 and 369 Hz, respectively, and lie within the expected range.[15] As a result of the ^2J(PF) and long range ^4J(PF) coupling with values of 55 Hz and of 2 Hz, the fluorine and the phosphorus NMR spectra show the magnetic nonequivalence of the two P(CF$_3$)$_2$ units. This allows the determination of the ^2J(PP) coupling via the calculation of the center signal as an [A$_6$X]$_2$ (A$_6$A'$_6$XX') spin system. On the knowledge that ^2J(PF) couplings - in contrast to the always negative ^1J(PF) couplings - are always positive,[16] the long range ^4J(PF) couplings can be assigned with a positive sign with respect to the interpretation of the experimental spectra as [A$_6$X]$_2$ spin systems. In general, for spin systems based on a [AX]$_2$ system, only the relative signs of the AX and AX' couplings are determinable.[17]

The magnitude of the ^2J(PP) couplings of 94 and 101 Hz for the C_2 symmetric chromium and tungsten compound, respectively, can be compared only with the ^2J(PP) couplings of the nonsymmetric compounds [Ag(μ-PR$_2$)M(CO)$_5$(μ-PR'$_2$)M'(CO)$_5$]$^-$ (R, R' = Ph, Cyhex and M, M' = Cr, Mo, W) with values of 130-150 Hz. These nonsymmetric compounds were obtained as a mixture via a synproportionation of the corresponding symmetric compounds.[4]

The determined ^2J(PP) coupling of 101 Hz for the tungsten derivative, [Ag{(μ-P(CF$_3$)$_2$)W(CO)$_5$}$_2$]$^-$, matches exactly with the value obtained via the calculation of the

tungsten satellites of the fluorine decoupled phosphorus spectrum. The isotopomer of the W-P-Ag-P-W unit with one NMR active ^{183}W isotope, gives rise to a magnetic nonequivalence of the two phosphorus atoms. The theoretical chemical difference of the two phosphorus atoms, caused by the isotopic shift, can be neglected in this case. The calculation of the two resulting $[A]_2MX$ (AA'MX) spin systems with A = P; M = ^{183}W and X = ^{109}Ag and ^{107}Ag allows the determination of the ^2J(PP) and the ^1J(WP) coupling constants. As a result of the ^3J(WP) coupling with a value close to zero it is not possible to determine the relative signs of the ^2J(PP) and the ^1J(WP) coupling constants via the iteration of the obtained experimental ^{31}P{^{19}F} NMR spectrum.

The highest vibrational CO valence mode of the series $[W(CO)_5PH(CF_3)_2]$,[6] $[Ag\{(\mu-P(CF_3)_2)W(CO)_5\}_2]$ and $[W\{P(CF_3)_2\}(CO)_5]^-$ [18] describes the expected shift to lower frequencies with about 2093, 2072 and 2065 cm^{-1}, respectively. These findings can be explained by the increasing electron density at the phosphorus atoms which causes a reduced π-back-bonding effect for the W-P bonds while the π-back-bonding effect for the W-C bonds increases in the opposite manner.

The highest CO valence mode of a comparable mercury complex $[Hg\{(\mu-P(CF_3)_2)W(CO)_5\}_2]\cdot2$ DMF,[3] located at 2081 cm^{-1} might be interpreted in terms of a stronger Lewis-acidity of the mercury center in comparison to the silver center in $[Ag\{(\mu-P(CF_3)_2)W(CO)_5\}_2]^-$.

Unfortunately, the reaction of $[K(18$-crown$-6)][Ag(CN)_2]$ with $HP(C_6F_5)_2$ did not yield a uniform product so far, while the reaction with $[W(CO)_5PH(C_6F_5)_2]$ allows the isolation of the comparable trinuclear product, $[K(18$-crown$-6)][Ag\{(\mu-P(C_6F_5)_2)W(CO)_5\}_2]$ in a 50% yield after recrystallization from a diethyl ether hexane mixture as a bright yellow powder.

$$[Ag(CN)_2]^- + 2 [W(CO)_5PH(C_6F_5)_2] \longrightarrow 2 HCN + [Ag\{(\mu\text{-}P(C_6F_5)_2)W(CO)_5\}_2]^- \quad (7)$$

$$cat = [K(18\text{-crown-6})]^+$$
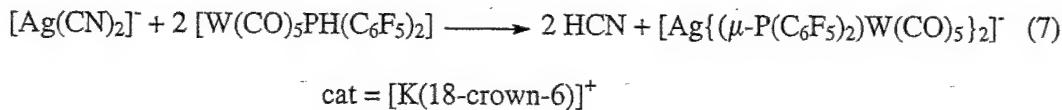
The experimental first order ^{31}P NMR spectrum of the trinuclear ion is shown in Fig. 4, showing a $^3J(PF)$ and a $^4J(PF)$ coupling of 9.0 and 8.4 Hz respectively. The coupling constants are determined via an iteration of the experimental spectrum (bottom trace of Figure 2).

As a result of the chemically as well as the magnetically equivalence of the two $P(C_6F_5)_2$ units, the $^2J(PP)$ coupling constant can not be determined from the fluorine decoupled phosphorus spectrum. In the case of the fluorine decoupled phosphorus spectrum, the tungsten satellites could be resolved (upper trace of Fig. 5). As already discussed for the CF_3 derivative, the isotopomer of the W-P-Ag-P-W unit with one NMR active ^{183}W isotope, gives rise to a magnetic nonequivalence of the two phosphorus atoms. The tungsten satellites are therefore a combination of two [A]$_2$MX spin systems (A = P, X = ^{183}W, M = ^{109}Ag and ^{107}Ag) one for each NMR active silver isotope. The calculation and iteration of the two spin systems allows the determination of the $^2J(PP)$ coupling constant to 142.4 Hz.

Only four lines for the isotopomer of the W-P-Ag-P-W unit with two NMR active ^{183}W isotopes could be assigned in the experimental fluorine decoupled ^{31}P NMR spectrum via the calculation of two sets of an [AX]$_2$M spin system. Two of them are marked with an arrow in the enlargement of Fig. 5.

The highest infrared CO valence mode of the trinuclear anion, $[Ag\{(\mu\text{-}P(C_6F_5)_2)M(CO)_5\}_2]^-$, located at 2066 cm^{-1} is shifted by 12 cm^{-1} to higher frequencies and by 18 cm^{-1} to lower frequencies in comparison to $[W\{P(C_6F_5)_2\}(CO)_5]^-$ and $[W(CO)_5PH(C_6F_5)_2]$, respectively and describes therefore the weaker and stronger, respectively, π-acidity of the comparable phosphorus moieties. The highest CO valence mode of $[Ag\{(\mu\text{-}$

- 17 -

$P(C_6F_5)_2)M(CO)_5\}_2]^-$, is shifted only 6 cm^{-1} to lower frequencies with respect to the comparable CF$_3$ derivative, $[Ag\{(\mu\text{-}P(CF_3)_2)M(CO)_5\}_2]^-$.

Supporting Information Available: Crystallographic file in CIF format for [K(18-crown-6)][Ag$\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2$]. This material is available free of charge via the Internet at http://pubs.acs.org.

References

(1) Grobe, J.; Haubold, R.; *Z. Anorg. Allg. Chem.,* **1986,** *534,* 121.

(2) Hoge, B.; Thösen, C.; Pantenburg, I. *Inorg. Chem.* **2001,** *40,* 3084-3088.

(3) Hoge, B.; Herrmann, T.; Thösen, C.; Pantenburg, I. *Inorg. Chem.* **2003** in press

(4) Odendorf, D.; Peringer, P. *Z. Naturforsch.* **1986,** *41b,* 79-81.

(5) Hoge, B.; Herrmann, T.; Thösen, C.; Pantenburg, I. *Inorg. Chem.* **2003,** *42,* 3623-3632.

(6) Grobe, J.; Le Van, D.; Meyering, W. *Z. Anorg. Chem.* **1990,** *586,* 149-158.

(7) Perrin, D. D.; Armarego, W. L. F.; Perrin,D. R. *Purification of Laboratory Chemicals;* Pergamon Press: Oxford, England, 1980.

(8) Budzelaar, P. H. M.; *gNMR Version 4.1.* Cherwell Scientific, Oxford, UK, 1998.

(9) Hoge, B.; Thösen, C. *Inorg. Chem.,* **2001,** *40,* 3113-3116.

(10) Grobe, J.; Demuth, R. *Angew. Chem.,* **1972,** *84,* 1153.

(11) Mahler, W.; Burg, A. B. *J. Am. Chem. Soc.* **1958,** *80,* 6161-6167.

(12) Albrand, J. P.; Cogne, A.; Robert, J. B. *J. Am. Chem. Soc.* **1978,** *100,* 2600-2604.

(13) Fluck, E.; Issleib, K. *Z. Anorg. Allg. Chem.* **1965,** *339,* 274-280.

(14) Eisenmann, J.; Fenske, D.; Simon, F. *Z. Anorg. Allg. Chem.* **1995,** *621,* 1681-1688; Eichhöfer, A.; Eisenmann, J.; Fenske, D.; Simon, F. *Z. Anorg. Allg. Chem.* **1993,** *619,* 1360-1368.

(15) Odendorf, D.; Peringer, P. *J. Organomet. Chem.* **1987,** *326,* 375-380; Odendorf, D.; Peringer, P. *J. Organomet. Chem.* **1986,** *299,* 127-130.

(16) Berger, S.; Braun, S.; Kalinowski, H.-O. *NMR-Spektroskopie von Nichtmetallen,* Georg Thieme Verlag Stuttgart, New York, 1993, pp. 147.

(17) Günther, H. *NMR-Spektroskopie,* Georg Thieme Verlag Stuttgart, New York, 1992, 3. ed., p. 175.

(18) Hoge, B.; Thösen, C.; Herrmann, T.; Pantenburg, I. *Inorg. Chem.* **2003,** *42,* 3633-3641.

Table 1. Crystal Data and Structure Refinement Parameters for [K(18-crown-6)][Ag{μ-P(CF$_3$)$_2$Cr(CO)$_5$}$_2$]

empirical formula	$C_{26}H_{24}AgCr_2F_{12}KO_{16}P_2$
formula mass	1133.36
T [K]	170(2)
space group	Fdd2 (no. 43)
a [pm]	2970.2(6)
b [pm]	1584.5(3)
c [pm]	1787.0(4)
V [nm^3]	8.41(3)
Z	8
ρ_{calc} [g cm^{-3}]	1.79
μ [mm^{-1}]	1.26
R indexes [$I>2\sigma I$]	R1 = 0.0280, $wR2$ = 0.0516
R indexes (all data)	R1 = 0.0359, $wR2$ = 0.0530
goodness of fit (S$_{obs}$)	1.014
Flack's parameter	-0.04(2)

R1 = ($\Sigma(F_o-Fc)/F_o$), $wR2$ = [$\Sigma(w(F_o-Fc)^2/wF_o^2$)

Table 2: Selected Bond Distances and Bond Angles for [K(18-crown-6)][Ag{μ-P(CF$_3$)$_2$Cr(CO)$_5$}$_2$]

Bond lengths (pm)

Ag(1)-P(1)	239.07(10)	Cr(1)-C(6)	189.2(5)
Ag(1)-P(1)^a	239.07(10)	Cr(1)-C(3)	189.3(5)
Cr(1)-C(7)	183.6(5)	Cr(1)-C(4)	190.7(6)
Cr(1)-C(5)	188.4(5)	Cr(1)-P(1)	238.90(13)

Bond Angles (°)

P(1)-Ag(1)-P(1)^a	169.17(6)	Cr(1)-P(1)-Ag(1)	119.68(4)
C(2)-P(1)-C(1)	95.1(2)	C(7)-Cr(1)-P(1)	177.68(17)
C(2)-P(1)-Cr(1)	111.50(16)	C(5)-Cr(1)-P(1)	89.95(14)
C(1)-P(1)-Cr(1)	114.95(15)	C(6)-Cr(1)-P(1)	92.06(15)
C(2)-P(1)-Ag(1)	107.31(15)	C(3)-Cr(1)-P(1)	87.36(13)
C(1)-P(1)-Ag(1)	105.35(15)	C(4)-Cr(1)-P(1)	92.36(14)

a = 3/2 − x, ½ - y, z

Table 3. NMR Spectroscopic Data for Phosphanide Derivatives and $(PCF_3)_4$[a]

	phosphanide unit			phosphane unit			
	$\delta(^{31}P)$	$\delta(^{19}F)$	$^2J(PF)$	$\delta(^{31}P)$	$\delta(^{19}F)$	$^2J(PF)$	$^1J(PP)$
$P(CF_3)_2^{-}$ [b]	-1.9	-31.4	47.2	-	-	-	-
$[Ag\{P(CF_3)_2\}_2]^{-}$ [c]	2.8	-39.4	57.9	-	-	-	-
$(PCF_3)_4$ [d]	-	-	-	-74.8	-51	[e]	100.6
$P(CF_3)P(CF_3)_2^{-}$ [f]	-92.0	-26.6	39.2	35.5	-51.4	62.0	339.7
$[Ag\{P(CF_3)P(CF_3)_2\}_2]^{-}$ [f]	-79.5	-32.1	44.7	21.9	-53.0	65.9	286.7
$P(C_2H_5)P(C_2H_5)(C_6H_5)^{-}$ [g]	-112.8	-	-	-17.2	-	-	396

[a] Chemical shifts in ppm; coupling constants in Hz.
[b] Reference 9.
[c] See Experimental Section.
[d] Reference 12.
[e] Not resolved.
[f] THF; 243 K.
[g] Reference 13.

Figure Legend

Figure 1. Simplified central projection of the C_2 symmetric $[Ag\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2]^-$ ion in the compound $[K(18\text{-crown-6})][Ag\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2]$ showing the atom numbering scheme of the asymmetric unit and thermal ellipsoids (40%). Selected bond lengths (pm) and angles (deg): Ag(1)-P(1) 239.1(1), P(1)-Cr(1) 238.9(1), P(1)-C(1) 187.5(4), P(1)-C(2) 187.5(5), P(1)-Ag(1)-P(1') 169.17(6), C(1)-P(1)-C(2) 95.1(2), Cr(1)-P(1)-Ag(1) 119.68(4).

Figure 2. Figure shown an ORTEP plot for the two components of the disordered 18-crown-6 ring. The thermal ellipsoids are at 30% probability level.

Figure 3. Crystal packing diagram of $[K(18\text{-crown-6})][Ag\{(\mu\text{-}P(CF_3)_2)Cr(CO)_5\}_2]$ viewed along the c-axis with the 18-crown-6 ring omitted for figure clarity. The vertical K…O/F and the horizontal Ag…O zig-zag polymeric chains run along the b- and a- axis, respectively.

Figure 4. Experimental (top) and calculated (bottom) ^{31}P NMR spectrum (81.01 MHz) of $[Ag\{(\mu\text{-}P(C_6F_5)_2)W(CO)_5\}_2]^-$.

Figure 5. Experimental (top) and calculated (bottom) ^{31}P{^{19}F} NMR spectrum (202.4 MHz) of $[Ag\{(\mu\text{-}P(C_6F_5)_2)W(CO)_5\}_2]^-$ (compare text).

Figure 1
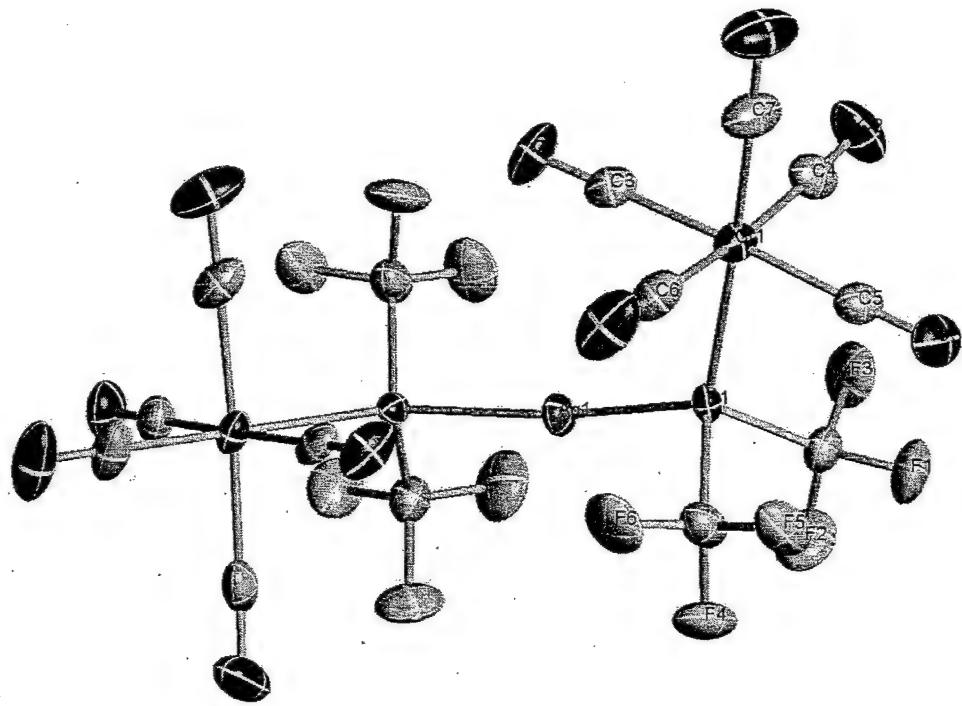
Figure 2
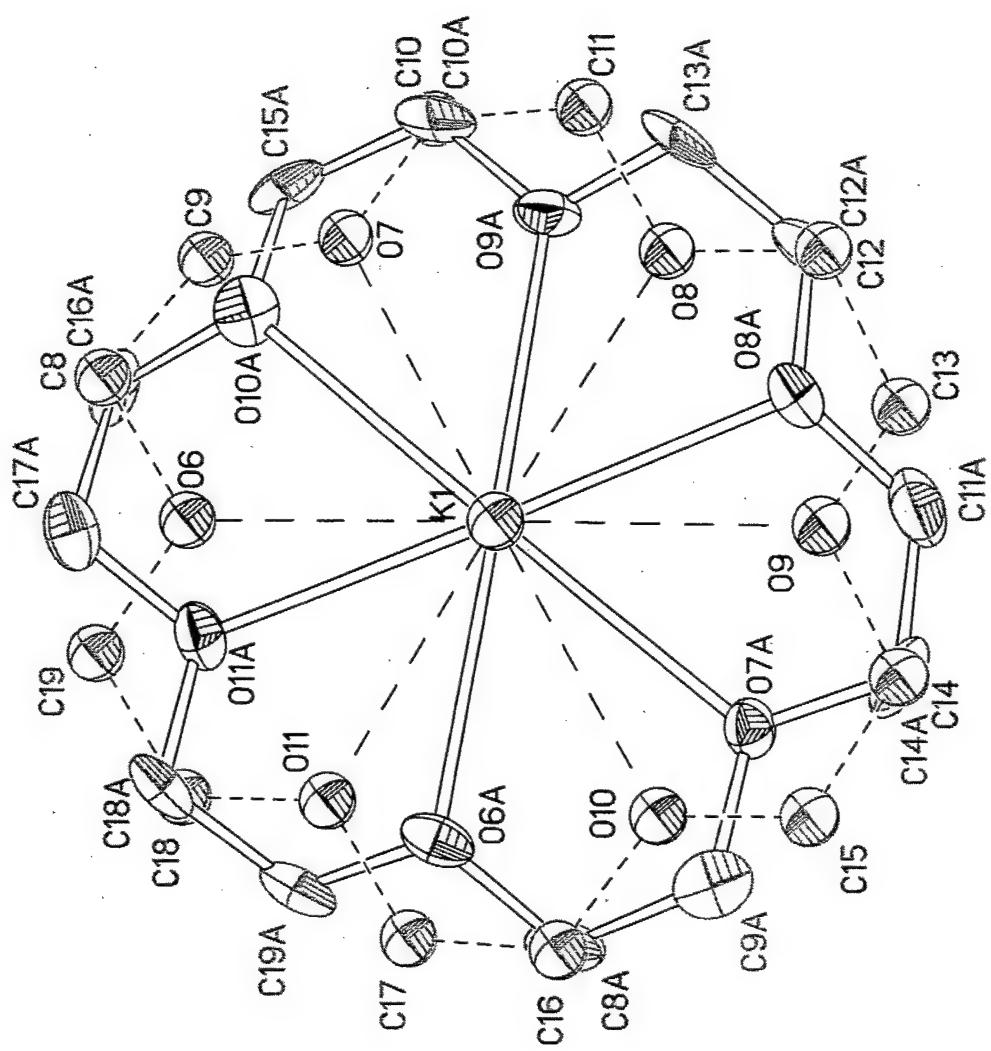
Figure 3
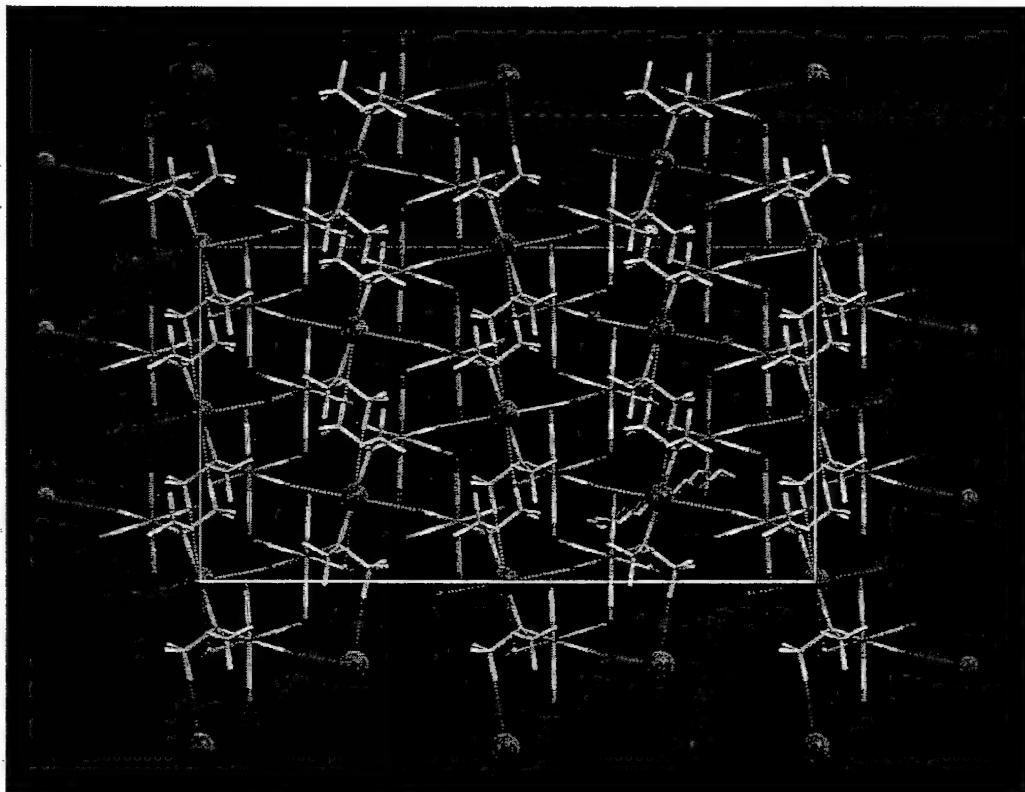
Packing along the c-axis showing the vertical K...O/F and the horizontal Ag...O chains

Figure 4
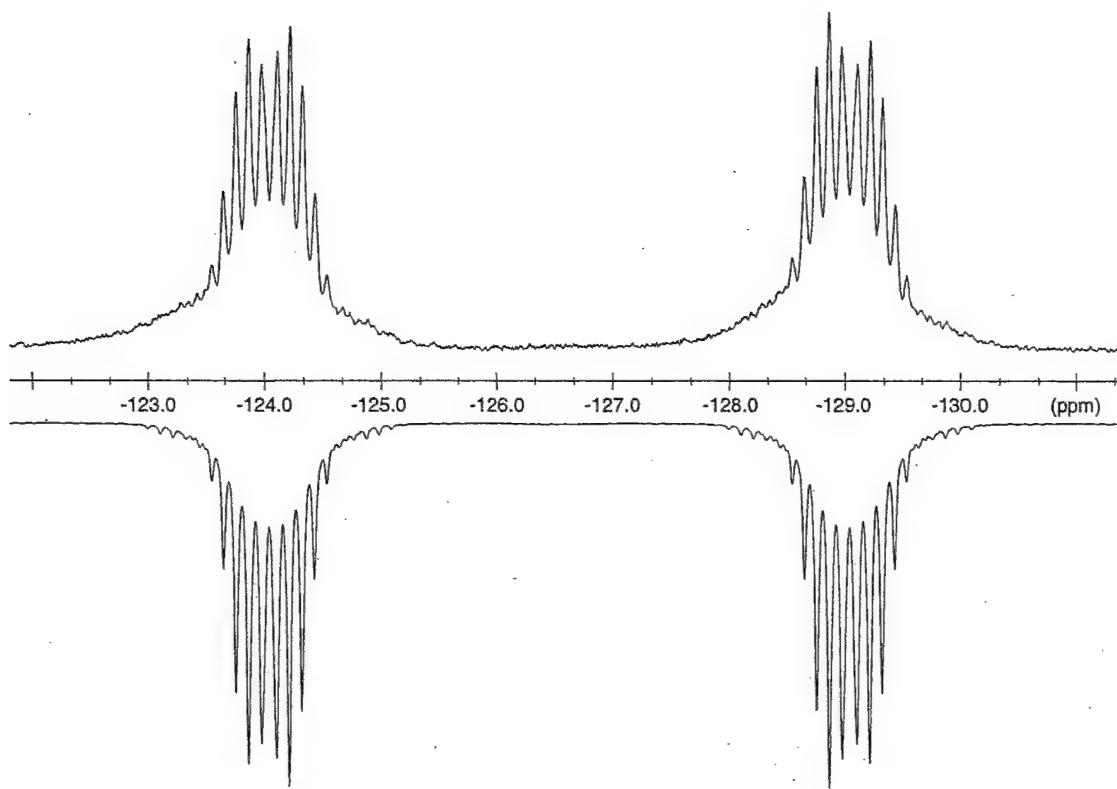
Figure 5
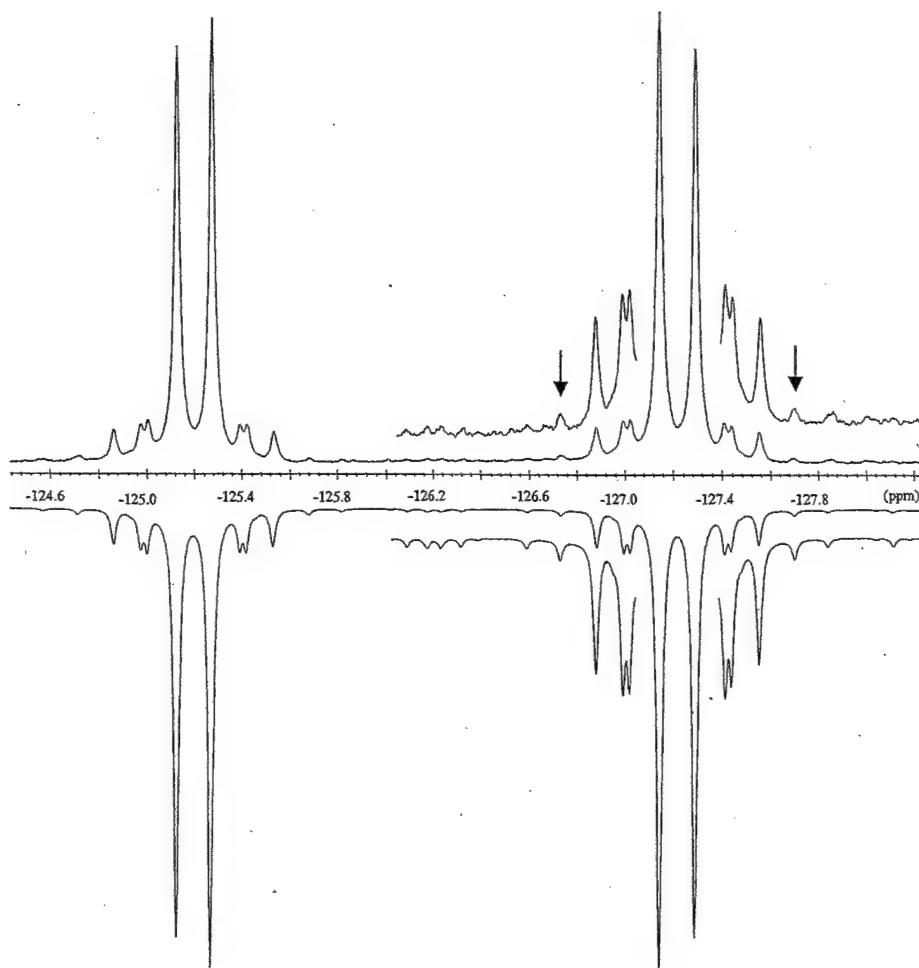
data_crown

_publ_contact_author_name   'Dr. Berthold Hoge'

_publ_contact_author_address

;Institut f\"ur Anorganische Chemie

Universit\"at zu K\"oln

Greinstr. 6

D-50939 K\"oln, Germany

;

_publ_contact_author_email   ': b.hoge@uni-koeln.de'

_publ_contact_author_fax   049-221-470-5196

_audit_creation_method   SHELXL-97

_chemical_name_systematic   ?

_chemical_name_common   '18-Crown-6-potassium

bis{pentacarbonyl-\µ-bis(trifluoromethyl)phosphanido}argentate(I)

'

_chemical_melting_point   ?

_chemical_formula_moiety   'C26 H24 Ag Cr2 F12 K O16 P2'

_chemical_formula_sum   'C26 H24 Ag Cr2 F12 K O16 P2'

_chemical_formula_weight   1133.36

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

_atom_type_scat_source

C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

H H 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

O O 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

F F 0.0171 0.0103 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

P P 0.1023 0.0942 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

K K 0.2009 0.2494 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

Cr Cr 0.3209 0.6236 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

Ag Ag -0.8971 1.1015 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting   orthorhombic

_symmetry_space_group_name_H-M Fdd2

_symmetry_Int_Tables_number   43

loop_

_symmetry_equiv_pos_as_xyz

'x, y, z'

'-x, -y, z'

'x+1/4, -y+1/4, z+1/4'

'-x+1/4, y+1/4, z+1/4'

'x, y+1/2, z+1/2'

'-x, -y+1/2, z+1/2'

'x+1/4, -y+3/4, z+3/4'

'-x+1/4, y+3/4, z+3/4'

'x+1/2, y, z+1/2'

'-x+1/2, -y, z+1/2'

'x+3/4, -y+1/4, z+3/4'

'-x+3/4, y+1/4, z+3/4'

'x+1/2, y+1/2, z'

'-x+1/2, -y+1/2, z'

'x+3/4, -y+3/4, z+1/4'

'-x+3/4, y+3/4, z+1/4'

_cell_length_a   29.702(6)

_cell_length_b   15.845(3)

_cell_length_c   17.870(4)

_cell_angle_alpha   90.00

_cell_angle_beta   90.00

_cell_angle_gamma   90.00

_cell_volume   8410(3)

_cell_formula_units_Z   8

_cell_measurement_temperature   170(2)

_cell_measurement_reflns_used   ?

_cell_measurement_theta_min   ?

_cell_measurement_theta_max   ?

_exptl_crystal_description   Columns

_exptl_crystal_colour   Colorless

_exptl_crystal_size_max   0.25

_exptl_crystal_size_mid   0.20

_exptl_crystal_size_min   0.05

_exptl_crystal_density_meas   ?

_exptl_crystal_density_diffrn   1.790

_exptl_crystal_density_method   'not measured'

_exptl_crystal_F_000   4480

_exptl_absorpt_coefficient_mu   1.260

_exptl_absorpt_correction_type   "numerical"

_exptl_absorpt_correction_T_min 0.4306

_exptl_absorpt_correction_T_max 0.2552

_exptl_absorpt_process_details ?

_exptl_special_details

;

?

;

_diffrn_ambient_temperature   170(2)

_diffrn_radiation_wavelength   0.71073z

_diffrn_source   'fine-focus sealed tube'

```
_diffrn_radiation_type            MoK \a
_diffrn_radiation_probe           x-ray
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   ?
_diffrn_measurement_method        ?
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         <1%
_diffrn_reflns_number             16303
_diffrn_reflns_av_R_equivalents   0.0559
_diffrn_reflns_av_sigmal/netl     0.0422
_diffrn_reflns_limit_h_min        -33
_diffrn_reflns_limit_h_max        33
_diffrn_reflns_limit_k_min        -18
_diffrn_reflns_limit_k_max        18
_diffrn_reflns_limit_l_min        -20
_diffrn_reflns_limit_l_max        20
_diffrn_reflns_theta_min          2.66
_diffrn_reflns_theta_max          24.13
_reflns_number_total              3310
_reflns_number_gt                 2866
_reflns_threshold_expression      >2sigma(I)


_computing_data_collection        ?
_computing_cell_refinement        ?
_computing_data_reduction         ?
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'SHELXTL V6.10'
_computing_publication_material   ?
```

_refine_special_details

;

Refinement of F^2 against ALL reflections. The weighted R-factor wR and

goodness of fit S are based on F^2, conventional R-factors R are based

on F, with F set to zero for negative F^2. The threshold expression of

$F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is

not relevant to the choice of reflections for refinement. R-factors based

on F^2 are statistically about twice as large as those based on F, and R-

factors based on ALL data will be even larger.

;

_refine_ls_structure_factor_coef Fsqd

_refine_ls_matrix_type   full

_refine_ls_weighting_scheme   calc

_refine_ls_weighting_details

'calc w=1/[\s^2^(Fo^2^)+(0.0237P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'

_atom_sites_solution_primary   direct

_atom_sites_solution_secondary difmap

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment   mixed

_refine_ls_extinction_method   none

_refine_ls_extinction_coef   ?

_refine_ls_abs_structure_details 'Flack H D (1983), Acta Cryst. A39, 876-881'

_refine_ls_abs_structure_Flack -0.04(2)

_refine_ls_number_reflns   3310

_refine_ls_number_parameters   353

_refine_ls_number_restraints   103

_refine_ls_R_factor_all   0.0359

_refine_ls_R_factor_gt   0.0280

_refine_ls_wR_factor_ref   0.0530

_refine_ls_wR_factor_gt   0.0516

_refine_ls_goodness_of_fit_ref 1.014

_refine_ls_restrained_S_all   0.999

_refine_ls_shift/su_max   0.001

_refine_ls_shift/su_mean   0.000

loop_

_atom_site_label

_atom_site_type_symbol

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_symmetry_multiplicity

_atom_site_calc_flag

_atom_site_refinement_flags

_atom_site_disorder_assembly

_atom_site_disorder_group

Ag1 Ag 0.7500 0.2500 0.81911(2) 0.03332(12) Uani 1 2 d S . .

Cr1 Cr 0.67165(2) 0.42727(4) 0.92781(4) 0.03394(17) Uani 1 1 d . . .

K1 K 0.7500 0.7500 0.84286(6) 0.0452(4) Uani 1 2 d S . .

P1 P 0.72438(3) 0.39232(6) 0.83174(6) 0.0258(2) Uani 1 1 d . . .

F1 F 0.70156(10) 0.50921(18) 0.72701(15) 0.0629(8) Uani 1 1 d . . .

F2 F 0.73728(11) 0.4043(2) 0.68172(14) 0.0760(9) Uani 1 1 d . . .

F3 F 0.66885(11) 0.3885(2) 0.71580(15) 0.0682(9) Uani 1 1 d . . .

F4 F 0.80336(10) 0.4554(2) 0.77720(19) 0.0690(9) Uani 1 1 d . . .

F5 F 0.76565(8) 0.54429(16) 0.84344(19) 0.0679(9) Uani 1 1 d . . .

F6 F 0.80000(10) 0.4409(2) 0.89641(18) 0.0708(10) Uani 1 1 d . . .

O1 O 0.67650(13) 0.2433(2) 0.97699(17) 0.0594(9) Uani 1 1 d . . .

O2 O 0.59005(12) 0.3801(2) 0.8319(3) 0.0750(11) Uani 1 1 d . . .

O3 O 0.67056(12) 0.6099(2) 0.8741(2) 0.0572(9) Uani 1 1 d . . .

O4 O 0.74559(16) 0.4704(3) 1.0389(2) 0.0793(13) Uani 1 1 d . . .

O5 O 0.60611(15) 0.4630(2) 1.0525(2) 0.0841(14) Uani 1 1 d . . .

C1 C 0.70754(16) 0.4258(3) 0.7351(2) 0.0387(11) Uani 1 1 d . . .

C2 C 0.77552(15) 0.4615(3) 0.8360(3) 0.0452(11) Uani 1 1 d . . .

C3 C 0.67456(16) 0.3129(3) 0.9581(2) 0.0388(11) Uani 1 1 d . . .

C4 C 0.62153(18) 0.3992(3) 0.8660(3) 0.0473(12) Uani 1 1 d . . .

C5 C 0.67095(16) 0.5400(3) 0.8944(2) 0.0413(11) Uani 1 1 d . . .

C6 C 0.71823(19) 0.4538(3) 0.9961(3) 0.0499(13) Uani 1 1 d . . .

C7 C 0.63164(18) 0.4500(3) 1.0035(3) 0.0561(14) Uani 1 1 d . . .

O6 O 0.8080(3) 0.7159(5) 0.9588(4) 0.0435(18) Uani 0.50 1 d PD A -1

O7 O 0.8322(2) 0.6736(4) 0.8124(4) 0.0396(14) Uani 0.50 1 d PD A -1

O8 O 0.7789(3) 0.7377(5) 0.6962(5) 0.0560(19) Uani 0.50 1 d PD A -1

O9 O 0.6878(3) 0.7756(5) 0.7276(4) 0.0532(19) Uani 0.50 1 d PD A -1

O10 O 0.6654(3) 0.8277(5) 0.8722(4) 0.049(2) Uani 0.50 1 d PD A -1

O11 O 0.7194(3) 0.7651(5) 0.9886(6) 0.0470(18) Uani 0.50 1 d PD A -1

C8 C 0.8436(6) 0.6582(12) 0.9454(10) 0.058(6) Uani 0.50 1 d PDU A -1

H8A H 0.8322 0.6008 0.9445 0.070 Uiso 0.50 1 calc PR A -1

H8B H 0.8661 0.6624 0.9845 0.070 Uiso 0.50 1 calc PR A -1

C9 C 0.8636(5) 0.6801(8) 0.8719(6) 0.054(3) Uani 0.50 1 d PDU A -1

H9A H 0.8751 0.7374 0.8740 0.065 Uiso 0.50 1 calc PR A -1

H9B H 0.8888 0.6428 0.8620 0.065 Uiso 0.50 1 calc PR A -1

C10 C 0.8504(7) 0.6865(16) 0.7398(7) 0.054(6) Uani 0.50 1 d PDU A -1

H10A H 0.8737 0.6451 0.7299 0.065 Uiso 0.50 1 calc PR A -1

H10B H 0.8637 0.7423 0.7365 0.065 Uiso 0.50 1 calc PR A -1

C11 C 0.8137(4) 0.6780(9) 0.6844(8) 0.065(4) Uani 0.50 1 d PDU A -1

H11A H 0.8259 0.6855 0.6345 0.078 Uiso 0.50 1 calc PR A -1

H11B H 0.8012 0.6215 0.6874 0.078 Uiso 0.50 1 calc PR A -1

C12 C 0.7436(6) 0.737(2) 0.6426(5) 0.102(8) Uani 0.50 1 d PDU A -1

H12A H 0.7301 0.6813 0.6412 0.123 Uiso 0.50 1 calc PR A -1

H12B H 0.7560 0.7487 0.5934 0.123 Uiso 0.50 1 calc PR A -1

C13 C 0.7082(6) 0.8002(13) 0.6596(5) 0.081(5) Uani 0.50 1 d PDU A -1

H13A H 0.7214 0.8559 0.6645 0.097 Uiso 0.50 1 calc PR A -1

H13B H 0.6860 0.8016 0.6198 0.097 Uiso 0.50 1 calc PR A -1

C14 C 0.6516(6) 0.8306(16) 0.7440(10) 0.068(8) Uani 0.50 1 d PDU A -1

H14A H 0.6286 0.8248 0.7057 0.082 Uiso 0.50 1 calc PR A -1

H14B H 0.6623 0.8884 0.7432 0.082 Uiso 0.50 1 calc PR A -1

C15 C 0.6314(3) 0.8123(7) 0.8183(7) 0.055(3) Uani 0.50 1 d PDU A -1

H15A H 0.6056 0.8486 0.8272 0.067 Uiso 0.50 1 calc PR A -1

H15B H 0.6215 0.7540 0.8208 0.067 Uiso 0.50 1 calc PR A -1

C16 C 0.6500(5) 0.8154(13) 0.9465(8) 0.058(6) Uani 0.50 1 d PDU A -1

H16A H 0.6383 0.7585 0.9515 0.070 Uiso 0.50 1 calc PR A -1

H16B H 0.6257 0.8545 0.9568 0.070 Uiso 0.50 1 calc PR A -1

C17 C 0.6866(3) 0.8284(6) 1.0019(7) 0.051(3) Uani 0.50 1 d PDU A -1

H17A H 0.6998 0.8840 0.9959 0.061 Uiso 0.50 1 calc PR A -1

H17B H 0.6750 0.8236 1.0524 0.061 Uiso 0.50 1 calc PR A -1

C18 C 0.7523(9) 0.7670(10) 1.0447(4) 0.054(5) Uani 0.50 1 d PDU A -1

H18A H 0.7385 0.7578 1.0932 0.064 Uiso 0.50 1 calc PR A -1

H18B H 0.7671 0.8216 1.0453 0.064 Uiso 0.50 1 calc PR A -1

C19 C 0.7857(4) 0.6996(8) 1.0292(7) 0.053(3) Uani 0.50 1 d PDU A -1

H19A H 0.8077 0.6976 1.0692 0.063 Uiso 0.50 1 calc PR A -1

H19B H 0.7706 0.6453 1.0271 0.063 Uiso 0.50 1 calc PR A -1

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

Ag1 0.0348(2) 0.0239(2) 0.0413(2) 0.000 0.000 0.0070(2)

Cr1 0.0426(4) 0.0257(3) 0.0336(3) -0.0067(3) 0.0068(3) -0.0027(3)

K1 0.0349(7) 0.0776(10) 0.0230(7) 0.000 0.000 0.0074(8)

P1 0.0258(5) 0.0222(5) 0.0293(5) -0.0001(4) -0.0064(5) 0.0030(4)

F1 0.087(2) 0.0502(18) 0.0511(15) 0.0189(13) -0.0126(15) 0.0126(16)

F2 0.106(3) 0.090(2) 0.0322(14) -0.0044(15) 0.0173(17) 0.0119(19)

F3 0.073(2) 0.082(2) 0.0495(16) 0.0091(15) -0.0385(16) -0.0095(18)

F4 0.0509(19) 0.0607(19) 0.096(2) 0.0036(16) 0.0313(17) -0.0125(15)

F5 0.0453(16) 0.0303(14) 0.128(3) -0.0102(16) 0.0013(17) -0.0128(11)

F6 0.0388(17) 0.086(3) 0.087(2) -0.0036(19) -0.0297(17) -0.0117(17)

O1 0.102(3) 0.033(2) 0.0441(18) 0.0082(15) -0.0057(19) -0.0126(19)

O2 0.041(2) 0.065(2) 0.119(3) -0.009(3) -0.019(3) 0.0024(17)

O3 0.066(2) 0.033(2) 0.073(2) -0.0043(17) 0.012(2) 0.0095(17)

O4 0.116(4) 0.078(3) 0.0442(19) 0.0017(18) -0.029(2) -0.034(2)

O5 0.105(3) 0.057(2) 0.091(3) -0.021(2) 0.056(3) -0.012(2)

C1 0.049(3) 0.031(3) 0.036(2) 0.004(2) -0.001(2) 0.003(2)

C2 0.035(2) 0.038(3) 0.062(3) -0.005(2) 0.000(3) -0.0012(19)

C3 0.048(3) 0.044(3) 0.025(2) -0.010(2) 0.005(2) -0.010(2)

C4 0.044(3) 0.031(3) 0.066(3) -0.008(2) 0.012(3) 0.004(2)

C5 0.043(3) 0.038(3) 0.043(3) -0.008(2) 0.009(2) -0.001(2)

C6 0.072(4) 0.042(3) 0.035(3) -0.003(2) 0.000(3) -0.016(3)

C7 0.074(4) 0.036(3) 0.059(3) -0.012(2) 0.026(3) -0.009(2)

O6 0.057(6) 0.035(4) 0.038(4) 0.007(4) -0.017(4) -0.014(4)

O7 0.037(5) 0.046(3) 0.036(4) -0.001(3) 0.007(3) -0.004(3)

O8 0.066(5) 0.072(5) 0.030(4) 0.006(4) 0.007(4) 0.008(4)

O9 0.053(6) 0.063(5) 0.043(4) 0.005(4) -0.019(4) 0.009(4)

O10 0.030(5) 0.059(5) 0.059(6) -0.005(4) 0.002(4) -0.020(4)

O11 0.046(5) 0.060(5) 0.034(4) -0.008(4) 0.010(4) -0.009(4)

C8 0.072(14) 0.048(9) 0.054(9) 0.011(6) -0.026(7) 0.010(8)

C9 0.050(9) 0.038(6) 0.074(9) 0.001(6) -0.008(8) -0.009(6)

C10 0.060(14) 0.054(9) 0.049(9) 0.001(7) 0.043(7) -0.008(8)

C11 0.085(11) 0.072(9) 0.039(8) 0.003(8) 0.020(7) 0.005(8)

C12 0.108(14) 0.18(2) 0.022(5) 0.001(11) -0.025(11) 0.045(12)

C13 0.097(13) 0.130(14) 0.017(6) 0.011(8) -0.021(7) 0.017(9)

C14 0.029(11) 0.086(16) 0.090(13) -0.002(10) -0.027(9) 0.009(9)

C15 0.019(6) 0.053(6) 0.094(9) -0.006(7) -0.024(6) 0.009(4)

C16 0.019(7) 0.083(14) 0.072(11) -0.028(8) 0.013(6) 0.003(8)

C17 0.055(7) 0.053(7) 0.043(7) -0.007(7) 0.015(6) -0.018(6)

C18 0.071(8) 0.069(15) 0.021(3) -0.001(5) -0.005(7) -0.009(10)

C19 0.075(10) 0.050(7) 0.032(6) 0.010(6) -0.023(7) -0.013(6)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

Ag1 P1 2.3907(10) . ?

Ag1 P1 2.3907(10) 14_655 ?

Cr1 C7 1.836(5) . ?

Cr1 C5 1.884(5) . ?

Cr1 C6 1.892(5) . ?

Cr1 C3 1.893(5) . ?

Cr1 C4 1.907(6) . ?

Cr1 P1 2.3890(13) . ?

K1 O6 2.749(8) . ?

K1 O6 2.749(8) 14_665 ?

K1 O8 2.764(8) . ?

K1 O8 2.764(8) 14_665 ?

K1 O11 2.768(9) 14_665 ?

K1 O11 2.768(9) . ?

K1 O7 2.778(6) . ?

K1 O7 2.778(6) 14_665 ?

K1 O9 2.797(7) 14_665 ?

K1 O9 2.797(7) . ?

K1 O10 2.847(9) 14_665 ?

K1 O10 2.847(9) . ?

P1 C2 1.875(5) . ?

P1 C1 1.875(4) . ?

F1 C1 1.342(5) . ?

F2 C1 1.343(5) . ?

F3 C1 1.337(5) . ?

F4 C2 1.340(6) . ?

F5 C2 1.351(5) . ?

F6 C2 1.342(6) . ?

O1 C3 1.156(5) . ?

O2 C4 1.156(6) . ?

O3 C5 1.165(5) . ?

O4 C6 1.146(6) . ?

O5 C7 1.177(5) . ?

O6 C8 1.419(9) . ?

O6 C19 1.446(13) . ?

O7 C10 1.419(9) . ?

O7 C9 1.420(8) . ?

O8 C11 1.417(8) . ?

O8 C12 1.422(9) . ?

O9 C13 1.412(8) . ?

O9 C14 1.414(9) . ?

O10 C15 1.416(7) . ?

O10 C16 1.418(9) . ?

O11 C18 1.400(19) . ?

O11 C17 1.418(8) . ?

C8 C9 1.483(17) . ?

C10 C11 1.479(17) . ?

C12 C13 1.482(17) . ?

C14 C15 1.485(17) . ?

C16 C17 1.485(16) . ?

C18 C19 1.483(17) . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

P1 Ag1 P1 169.17(6) . 14_655 ?

C7 Cr1 C5 92.3(2) . . ?

C7 Cr1 C6 87.4(2) . . ?

C5 Cr1 C6 90.1(2) . . ?

C7 Cr1 C3 90.4(2) . . ?

C5 Cr1 C3 177.3(2) . . ?

C6 Cr1 C3 89.7(2) . . ?

C7 Cr1 C4 88.1(2) . . ?

C5 Cr1 C4 91.7(2) . . ?

C6 Cr1 C4 175.2(2) . . ?

C3 Cr1 C4 88.7(2) . . ?

C7 Cr1 P1 177.68(17) . . ?

C5 Cr1 P1 89.95(14) . . ?

C6 Cr1 P1 92.06(15) . . ?

C3 Cr1 P1 87.36(13) . . ?

C4 Cr1 P1 92.36(14) . . ?

O6 K1 O6 82.1(4) . 14_665 ?

O6 K1 O8 120.4(3) . . ?

O6 K1 O8 157.4(3) 14_665 . ?

O6 K1 O8 157.4(3) . 14_665 ?

O6 K1 O8 120.4(3) 14_665 14_665 ?

O8 K1 O8 37.1(3) . 14_665 ?

O6 K1 O11 21.28(18) . 14_665 ?

O6 K1 O11 60.9(3) 14_665 14_665 ?

O8 K1 O11 141.6(3) . 14_665 ?

O8 K1 O11 178.6(3) 14_665 14_665 ?

O6 K1 O11 60.9(3) . . ?

O6 K1 O11 21.28(18) 14_665 . ?

O8 K1 O11 178.6(3) . . ?

O8 K1 O11 141.6(3) 14_665 . ?

O11 K1 O11 39.7(4) 14_665 . ?

O6 K1 O7 60.8(2) . . ?

O6 K1 O7 141.6(2) 14_665 . ?

O8 K1 O7 60.7(2) . . ?

O8 K1 O7 96.7(2) 14_665 . ?

O11 K1 O7 81.9(2) 14_665 . ?

O11 K1 O7 120.7(2) . . ?

O6 K1 O7 141.6(2) . 14_665 ?

O6 K1 O7 60.8(2) 14_665 14_665 ?

O8 K1 O7 96.7(2) . 14_665 ?

O8 K1 O7 60.7(2) 14_665 14_665 ?

O11 K1 O7 120.7(2) 14_665 14_665 ?

O11 K1 O7 81.9(2) . 14_665 ?

O7 K1 O7 157.4(3) . 14_665 ?

O6 K1 O9 96.4(3) . 14_665 ?

O6 K1 O9 176.4(2) 14_665 14_665 ?

O8 K1 O9 24.0(2) . 14_665 ?

O8 K1 O9 61.1(3) 14_665 14_665 ?

O11 K1 O9 117.6(3) 14_665 14_665 ?

O11 K1 O9 157.2(3) . 14_665 ?

O7 K1 O9 38.0(2) . 14_665 ?

O7 K1 O9 120.0(2) 14_665 14_665 ?

O6 K1 O9 176.4(2) . . ?

O6 K1 O9 96.4(3) 14_665 . ?

O8 K1 O9 61.1(3) . . ?

O8 K1 O9 24.0(2) 14_665 . ?

O11 K1 O9 157.2(3) 14_665 . ?

O11 K1 O9 117.6(3) . . ?

O7 K1 O9 120.0(2) . . ?

O7 K1 O9 38.0(2) 14_665 . ?

O9 K1 O9 85.2(3) 14_665 . ?

O6 K1 O10 39.0(2) . 14_665 ?

O6 K1 O10 120.0(3) 14_665 14_665 ?

O8 K1 O10 82.5(2) . 14_665 ?

O8 K1 O10 118.6(2) 14_665 14_665 ?

O11 K1 O10 60.0(2) 14_665 14_665 ?

O11 K1 O10 98.9(2) . 14_665 ?

O7 K1 O10 21.92(17) . 14_665 ?

O7 K1 O10 179.2(2) 14_665 14_665 ?

O9 K1 O10 59.3(2) 14_665 14_665 ?

O9 K1 O10 141.4(2) . 14_665 ?

O6 K1 O10 120.0(3) . . ?

O6 K1 O10 39.0(2) 14_665 . ?

O8 K1 O10 118.6(2) . . ?

O8 K1 O10 82.5(2) 14_665 . ?

O11 K1 O10 98.9(2) 14_665 . ?

O11 K1 O10 60.0(2) . . ?

O7 K1 O10 179.2(2) . . ?

O7 K1 O10 21.92(17) 14_665 . ?

O9 K1 O10 141.4(2) 14_665 . ?

O9 K1 O10 59.3(2) . . ?

O10 K1 O10 158.8(3) 14_665 . ?

C2 P1 C1 95.1(2) . . ?

C2 P1 Cr1 111.50(16) . . ?

C1 P1 Cr1 114.95(15) . . ?

C2 P1 Ag1 107.31(15) . . ?

C1 P1 Ag1 105.35(15) . . ?

Cr1 P1 Ag1 119.68(4) . . ?

C2 F5 K1 172.7(3) . . ?

C5 O3 K1 133.4(3) . . ?

F3 C1 F1 107.1(4) . . ?

F3 C1 F2 105.7(4) . . ?

F1 C1 F2 105.1(4) . . ?

F3 C1 P1 110.0(3) . . ?

F1 C1 P1 114.4(3) . . ?

F2 C1 P1 114.0(3) . . ?

F4 C2 F6 106.2(4) . . ?

F4 C2 F5 106.3(4) . . ?

F6 C2 F5 106.0(4) . . ?

F4 C2 P1 115.2(3) . . ?

F6 C2 P1 109.2(3) . . ?

F5 C2 P1 113.3(3) . . ?

O1 C3 Cr1 179.6(5) . . ?

O2 C4 Cr1 176.3(5) . . ?

O3 C5 Cr1 179.7(5) . . ?

O4 C6 Cr1 178.2(5) . . ?

O5 C7 Cr1 178.7(5) . . ?

C8 O6 C19 112.0(12) . . ?

C8 O6 K1 117.8(8) . . ?

C19 O6 K1 113.8(8) . . ?

C10 O7 C9 115.1(13) . . ?

C10 O7 K1 116.8(9) . . ?

C9 O7 K1 113.5(8) . . ?

C11 O8 C12 115.6(17) . . ?

C11 O8 K1 114.5(8) . . ?

C12 O8 K1 114.3(8) . . ?

C13 O9 C14 109.6(15) . . ?

C13 O9 K1 112.9(9) . . ?

C14 O9 K1 116.0(8) . . ?

C15 O10 C16 112.6(12) . . ?

C15 O10 K1 115.5(7) . . ?

C16 O10 K1 113.4(7) . . ?

C18 O11 C17 110.1(14) . . ?

C18 O11 K1 116.5(9) . . ?

C17 O11 K1 116.4(8) . . ?

O6 C8 C9 107.3(13) . . ?

O7 C9 C8 112.5(15) . . ?

O7 C10 C11 108.6(15) . . ?

O8 C11 C10 112.1(15) . . ?

O8 C12 C13 112.3(17) . . ?

O9 C13 C12 107.2(14) . . ?

O9 C14 C15 111.9(15) . . ?

O10 C15 C14 106.6(13) . . ?

O10 C16 C17 111.7(12) . . ?

O11 C17 C16 107.0(13) . . ?

O11 C18 C19 108.5(11) . . ?

O6 C19 C18 109.9(13) . . ?

loop_

_geom_torsion_atom_site_label_1

_geom_torsion_atom_site_label_2

_geom_torsion_atom_site_label_3

_geom_torsion_atom_site_label_4

_geom_torsion

_geom_torsion_site_symmetry_1

_geom_torsion_site_symmetry_2

_geom_torsion_site_symmetry_3

_geom_torsion_site_symmetry_4

_geom_torsion_publ_flag

C7 Cr1 P1 C2 -111(4) . . . . ?

C5 Cr1 P1 C2 55.1(2) . . . . ?

C6 Cr1 P1 C2 -35.0(2) . . . . ?

C3 Cr1 P1 C2 -124.6(2) . . . . ?

C4 Cr1 P1 C2 146.8(2) . . . . ?

C7 Cr1 P1 C1 142(4) . . . . ?

C5 Cr1 P1 C1 -51.6(2) . . . . ?

C6 Cr1 P1 C1 -141.7(2) . . . . ?

C3 Cr1 P1 C1 128.7(2) . . . . ?

C4 Cr1 P1 C1 40.0(2) . . . . ?

C7 Cr1 P1 Ag1 15(4) . . . . ?

C5 Cr1 P1 Ag1 -178.56(15) . . . . ?

C6 Cr1 P1 Ag1 91.34(15) . . . . ?

C3 Cr1 P1 Ag1 1.72(14) . . . . ?

C4 Cr1 P1 Ag1 -86.90(15) . . . . ?

P1 Ag1 P1 C2 89.25(18) 14_655 . . . ?

P1 Ag1 P1 C1 -170.31(16) 14_655 . . . ?

P1 Ag1 P1 Cr1 -39.02(4) 14_655 . . . ?

O6 K1 F5 C2 -104(2) . . . . ?

O6 K1 F5 C2 179(100) 14_665 . . . ?

O8 K1 F5 C2 20(2) . . . . ?

O8 K1 F5 C2 55(2) 14_665 . . . ?

O11 K1 F5 C2 -124(2) 14_665 . . . ?

O11 K1 F5 C2 -161(2) . . . . ?

O7 K1 F5 C2 -39(2) . . . . ?

O7 K1 F5 C2 114(2) 14_665 . . . ?

O9 K1 F5 C2 -3(2) 14_665 . . . ?

O9 K1 F5 C2 78(2) . . . . ?

O10 K1 F5 C2 -65(2) 14_665 . . . ?

O10 K1 F5 C2 140(2) . . . . ?

O6 K1 O3 C5 -38.5(5) . . . . ?

O6 K1 O3 C5 -113.9(5) 14_665 . . . ?

O8 K1 O3 C5 89.5(4) . . . . ?

O8 K1 O3 C5 121.1(4) 14_665 . . . ?

O11 K1 O3 C5 -57.5(5) 14_665 . . . ?

O11 K1 O3 C5 -92.0(5) . . . . ?

O7 K1 O3 C5 24.2(5) . . . . ?

O7 K1 O3 C5 -179.5(5) 14_665 . . . ?

O9 K1 O3 C5 65.6(5) 14_665 . . . ?

O9 K1 O3 C5 141.1(4) . . . . ?

O10 K1 O3 C5 1.2(5) 14_665 . . . ?

O10 K1 O3 C5 -156.6(5) . . . . ?

C2 P1 C1 F3 -177.0(3) . . . . ?

Cr1 P1 C1 F3 -60.5(3) . . . . ?

Ag1 P1 C1 F3 73.5(3) . . . . ?

C2 P1 C1 F1 -56.5(4) . . . . ?

Cr1 P1 C1 F1 60.1(4) . . . . ?

Ag1 P1 C1 F1 -166.0(3) . . . . ?

C2 P1 C1 F2 64.5(4) . . . . ?

Cr1 P1 C1 F2 -178.9(3) . . . . ?

Ag1 P1 C1 F2 -45.0(4) . . . . ?

K1 F5 C2 F4 0(2) . . . . ?

K1 F5 C2 F6 113(2) . . . . ?

K1 F5 C2 P1 -127(2) . . . . ?

C1 P1 C2 F4 -50.4(4) . . . . ?

Cr1 P1 C2 F4 -169.7(3) . . . . ?

Ag1 P1 C2 F4 57.4(4) . . . . ?

C1 P1 C2 F6 -169.7(3) . . . . ?

Cr1 P1 C2 F6 70.9(3) . . . . ?

Ag1 P1 C2 F6 -61.9(3) . . . . ?

C1 P1 C2 F5 72.4(4) . . . . ?

Cr1 P1 C2 F5 -46.9(4) . . . . ?

Ag1 P1 C2 F5 -179.8(3) . . . . ?

C7 Cr1 C3 O1 -77(80) . . . . ?

C5 Cr1 C3 O1 96(80) . . . . ?

C6 Cr1 C3 O1 10(80) . . . . ?

C4 Cr1 C3 O1 -165(100) . . . . ?

P1 Cr1 C3 O1 102(100) . . . . ?

C7 Cr1 C4 O2 -38(6) . . . . ?

C5 Cr1 C4 O2 -130(6) . . . . ?

C6 Cr1 C4 O2 -19(8) . . . . ?

C3 Cr1 C4 O2 52(6) . . . . ?

P1 Cr1 C4 O2 140(6) . . . . ?

K1 O3 C5 Cr1 73(88) . . . . ?

C7 Cr1 C5 O3 54(88) . . . . ?

C6 Cr1 C5 O3 -34(88) . . . . ?

C3 Cr1 C5 O3 -120(100) . . . . ?

C4 Cr1 C5 O3 142(100) . . . . ?

P1 Cr1 C5 O3 -126(100) . . . . ?

C7 Cr1 C6 O4 -2(14) . . . . ?

C5 Cr1 C6 O4 91(14) . . . . ?

C3 Cr1 C6 O4 -92(14) . . . . ?

C4 Cr1 C6 O4 -21(15) . . . . ?

P1 Cr1 C6 O4 -179(100) . . . . ?

C5 Cr1 C7 O5 -174(100) . . . . ?

C6 Cr1 C7 O5 -84(23) . . . . ?

C3 Cr1 C7 O5 6(23) . . . . ?

C4 Cr1 C7 O5 95(23) . . . . ?

P1 Cr1 C7 O5 -8(27) . . . . ?

O6 K1 O6 C8 153.6(11) 14_665 . . . ?

O8 K1 O6 C8 -28.1(11) . . . . ?

O8 K1 O6 C8 -22.6(13) 14_665 . . . ?

O11 K1 O6 C8 156.1(13) 14_665 . . . ?

O11 K1 O6 C8 152.6(11) . . . . ?

O7 K1 O6 C8 -16.3(11) . . . . ?

O7 K1 O6 C8 167.9(11) 14_665 . . . ?

O9 K1 O6 C8 -29.8(11) 14_665 . . . ?

O9 K1 O6 C8 86(4) . . . . ?

O10 K1 O6 C8 -12.7(10) 14_665 . . . ?

O10 K1 O6 C8 163.5(11) . . . . ?

O6 K1 O6 C19 19.7(6) 14_665 . . . ?

O8 K1 O6 C19 -162.0(7) . . . . ?

O8 K1 O6 C19 -156.5(7) 14_665 . . . ?

O11 K1 O6 C19 22.2(7) 14_665 . . . ?

O11 K1 O6 C19 18.7(7) . . . . ?

O7 K1 O6 C19 -150.2(8) . . . . ?

O7 K1 O6 C19 34.0(8) 14_665 . . . ?

O9 K1 O6 C19 -163.7(7) 14_665 . . . ?

O9 K1 O6 C19 -48(4) . . . . ?

O10 K1 O6 C19 -146.6(8) 14_665 . . . ?

O10 K1 O6 C19 29.6(8) . . . . ?

O6 K1 O7 C10 -153.7(13) . . . . ?

O6 K1 O7 C10 -170.0(13) 14_665 . . . ?

O8 K1 O7 C10 14.6(13) . . . . ?

O8 K1 O7 C10 23.8(13) 14_665 . . . ?

O11 K1 O7 C10 -156.5(13) 14_665 . . . ?

O11 K1 O7 C10 -165.1(13) . . . . ?

O7 K1 O7 C10 19.5(13) 14_665 . . . ?

O9 K1 O7 C10 4.1(13) 14_665 . . . ?

O9 K1 O7 C10 30.4(13) . . . . ?

O10 K1 O7 C10 -159.8(14) 14_665 . . . ?

O10 K1 O7 C10 40(19) . . . . ?

O6 K1 O7 C9 -16.2(7) . . . . ?

O6 K1 O7 C9 -32.4(8) 14_665 . . . ?

O8 K1 O7 C9 152.1(8) . . . . ?

O8 K1 O7 C9 161.4(7) 14_665 . . . ?

O11 K1 O7 C9 -19.0(7) 14_665 . . . ?

O11 K1 O7 C9 -27.5(8) . . . . ?

O7 K1 O7 C9 157.1(7) 14_665 . . . ?

O9 K1 O7 C9 141.7(8) 14_665 . . . ?

O9 K1 O7 C9 167.9(7) . . . . ?

O10 K1 O7 C9 -22.2(7) 14_665 . . . ?

O10 K1 O7 C9 178(100) . . . . ?

O6 K1 O8 C11 29.1(10) . . . . ?

O6 K1 O8 C11 -155.3(8) 14_665 . . . ?

Q8 K1 O8 C11 -147.4(12) 14_665 . . . ?

O11 K1 O8 C11 31.5(10) 14_665 . . . ?

O11 K1 O8 C11 -175(10) . . . . ?

O7 K1 O8 C11 17.3(9) . . . . ?

O7 K1 O8 C11 -160.8(9) 14_665 . . . ?

O9 K1 O8 C11 33.2(8) 14_665 . . . ?

O9 K1 O8 C11 -147.1(9) . . . . ?

O10 K1 O8 C11 19.4(9) 14_665 . . . ?

O10 K1 O8 C11 -162.4(9) . . . . ?

O6 K1 O8 C12 165.7(15) . . . . ?

O6 K1 O8 C12 -18.7(17) 14_665 . . . ?

O8 K1 O8 C12 -10.8(15) 14_665 . . . ?

O11 K1 O8 C12 168.1(15) 14_665 . . . ?

O11 K1 O8 C12 -39(11) . . . . ?

O7 K1 O8 C12 153.9(16) . . . . ?

O7 K1 O8 C12 -24.2(15) 14_665 . . . ?

O9 K1 O8 C12 169.8(18) 14_665 . . . ?

O9 K1 O8 C12 -10.5(15) . . . . ?

O10 K1 O8 C12 156.0(16) 14_665 . . . ?

O10 K1 O8 C12 -25.7(16) . . . . ?

O6 K1 O9 C13 -139(4) . . . . ?

O6 K1 O9 C13 153.6(10) 14_665 . . . ?

O8 K1 O9 C13 -23.2(10) . . . . ?

O8 K1 O9 C13 -22.8(10) 14_665 . . . ?

O11 K1 O9 C13 159.0(10) 14_665 . . . ?

O11 K1 O9 C13 156.0(10) . . . . ?

O7 K1 O9 C13 -38.9(11) . . . . ?

O7 K1 O9 C13 134.3(11) 14_665 . . . ?

O9 K1 O9 C13 -23.1(10) 14_665 . . . ?

O10 K1 O9 C13 -45.0(11) 14_665 . . . ?

O10 K1 O9 C13 141.2(11) . . . . ?

O6 K1 O9 C14 93(4) . . . . ?

O6 K1 O9 C14 25.9(14) 14_665 . . . ?

O8 K1 O9 C14 -151.0(14) . . . . ?

O8 K1 O9 C14 -150.5(16) 14_665 . . . ?

O11 K1 O9 C14 31.2(16) 14_665 . . . ?

O11 K1 O9 C14 28.3(14) . . . . ?

O7 K1 O9 C14 -166.7(14) . . . . ?

O7 K1 O9 C14 6.5(13) 14_665 . . . ?

O9 K1 O9 C14 -150.8(14) 14_665 . . . ?

O10 K1 O9 C14 -172.7(14) 14_665 . . . ?

O10 K1 O9 C14 13.5(14) . . . . ?

O6 K1 O10 C15 -155.4(7) . . . . ?

O6 K1 O10 C15 -139.7(9) 14_665 . . . ?

O8 K1 O10 C15 36.0(9) . . . . ?

O8 K1 O10 C15 27.0(8) 14_665 . . . ?

O11 K1 O10 C15 -152.6(8) 14_665 . . . ?

O11 K1 O10 C15 -144.4(9) . . . . ?

O7 K1 O10 C15 10(19) . . . . ?

O7 K1 O10 C15 32.0(7) 14_665 . . . ?

O9 K1 O10 C15 46.1(9) 14_665 . . . ?

O9 K1 O10 C15 20.5(7) . . . . ?

O10 K1 O10 C15 -148.8(8) 14_665 . . . ?

O6 K1 O10 C16 -23.4(11) . . . . ?

O6 K1 O10 C16 -7.7(10) 14_665 . . . ?

O8 K1 O10 C16 168.0(10) . . . . ?

O8 K1 O10 C16 159.0(10) 14_665 . . . ?

O11 K1 O10 C16 -20.7(10) 14_665 . . . ?

O11 K1 O10 C16 -12.4(10) . . . . ?

O7 K1 O10 C16 142(19) . . . . ?

O7 K1 O10 C16 163.9(13) 14_665 . . . ?

O9 K1 O10 C16 178.1(10) 14_665 . . . ?

O9 K1 O10 C16 152.5(11) . . . . ?

O10 K1 O10 C16 -16.8(10) 14_665 . . . ?

O6 K1 O11 C18 14.8(9) . . . . ?

O6 K1 O11 C18 -162.4(13) 14_665 . . . ?

O8 K1 O11 C18 -141(11) . . . . ?

O8 K1 O11 C18 -168.2(8) 14_665 . . . ?

O11 K1 O11 C18 12.8(7) 14_665 . . . ?

O7 K1 O11 C18 26.1(9) . . . . ?

O7 K1 O11 C18 -155.7(9) 14_665 . . . ?

O9 K1 O11 C18 8.7(12) 14_665 . . . ?

O9 K1 O11 C18 -169.0(9) . . . . ?

O10 K1 O11 C18 24.1(9) 14_665 . . . ?

O10 K1 O11 C18 -154.3(9) . . . . ?

O6 K1 O11 C17 147.4(8) . . . . ?

O6 K1 O11 C17 -29.8(6) 14_665 . . . ?

O8 K1 O11 C17 -8(11) . . . . ?

O8 K1 O11 C17 -35.6(9) 14_665 . . . ?

O11 K1 O11 C17 145.4(10) 14_665 . . . ?

O7 K1 O11 C17 158.7(7) . . . . ?

O7 K1 O11 C17 -23.1(7) 14_665 . . . ?

O9 K1 O11 C17 141.3(7) 14_665 . . . ?

O9 K1 O11 C17 -36.4(8) . . . . ?

O10 K1 O11 C17 156.7(7) 14_665 . . . ?

O10 K1 O11 C17 -21.7(7) . . . . ?

C19 O6 C8 C9 179.4(11) . . . . ?

K1 O6 C8 C9 44.7(17) . . . . ?

C10 O7 C9 C8 -174.6(14) . . . . ?

K1 O7 C9 C8 47.1(12) . . . . ?

O6 C8 C9 O7 -60.8(16) . . . . ?

C9 O7 C10 C11 -179.8(12) . . . . ?

K1 O7 C10 C11 -43(2) . . . . ?

C12 O8 C11 C10 176.4(14) . . . . ?

K1 O8 C11 C10 -47.6(14) . . . . ?

O7 C10 C11 O8 60.1(19) . . . . ?

C11 O8 C12 C13 179.0(15) . . . . ?

K1 O8 C12 C13 43(2) . . . . ?

C14 O9 C13 C12 -176.0(14) . . . . ?

K1 O9 C13 C12 52.9(15) . . . . ?

O8 C12 C13 O9 -65(2) . . . . ?

C13 O9 C14 C15 -175.0(14) . . . . ?

K1 O9 C14 C15 -46(2) . . . . ?

C16 O10 C15 C14 178.0(12) . . . . ?

K1 O10 C15 C14 -49.7(11) . . . . ?

O9 C14 C15 O10 63.1(19) . . . . ?

C15 O10 C16 C17 177.7(11) . . . . ?

K1 O10 C16 C17 44.3(16) . . . . ?

C18 O11 C17 C16 -172.7(12) . . . . ?

K1 O11 C17 C16 51.9(10) . . . . ?

O10 C16 C17 O11 -64.0(15) . . . . ?

C17 O11 C18 C19 179.5(11) . . . . ?

K1 O11 C18 C19 -45.1(14) . . . . ?

C8 O6 C19 C18 173.7(14) . . . . ?

K1 O6 C19 C18 -49.7(12) . . . . ?

O11 C18 C19 O6 62.9(15) . . . . ?

_diffrn_measured_fraction_theta_max 0.989

_diffrn_reflns_theta_full   24.13

_diffrn_measured_fraction_theta_full 0.989

_refine_diff_density_max   0.531

_refine_diff_density_min   -0.306

_refine_diff_density_rms   0.062

loop_

_publ_author_name

_publ_author_address

'Hoge, Berthold'

;Institut f\"ur Anorganische Chemie

Universit\"at zu K\"oln

Greinstr. 6

D-50939 K\"oln, Germany

;

'Th\"osen, Christoph'

;Institut f\"ur Anorganische Chemie

Universit\"at zu K\"oln

Greinstr. 6

D-50939 K\"oln, Germany

;

'Herrmann, Tobias '

;Institut f\"ur Anorganische Chemie

Universit\"at zu K\"oln

Greinstr. 6

D-50939 K\"oln, Germany

;

'Vij, Ashwani'

;Air Force Research Laboratory, PRSP

Space and Missile Division

Edwards Air Force Base

Edwards CA 93536

661-275-6278 (Phone), 661-275-5471 (FAX)

ashwani.vij@edwards.af.mil

;

_ccdc_chemical_compound_source_recrystallisation 'hexane/diethyl ether'